IS 6921 (1973): Methods of sampling and test for lac and lac products [CHD 23: Lac, Lac Products and Polishes]

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IS : 6921 - 1973

# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR LAC AND LAC PRODUCTS

(Second Reprint SEPTEMBER 1990)

UDC 668.447.3 : 620.113



**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN 9, BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 10 *October* 1973

# AMENDMENT NO. 2 FEBRUARY 2007
# TO
# IS 6921 : 1973 METHODS OF SAMPLING AND TEST FOR LAC AND LAC PRODUCTS

(*Page* 56, *clause* 27) — Insert the following after clause 27:

## 28 UV AND VISIBLE SPECTRAL ANALYSIS FOR ASSERTION OF QUALITY OF SEEDLAC, SHELLAC AND BLEACHED LAC

### 28.1 General

Shellac (lac resin) contains polyhydroxyanthraquinone derivative pigments, of erythrolaccin group. The constituent acids of the resin also contain double bonds. These lead to characteristic absorption maxima in visible and UV range.

During the bleaching process, the chromophoric groups of the colouring pigments are destroyed, resulting in absence of absorption maxima in the visible region.

### 28.2 Outline of the Method

The method involves running of wavelength scan (absorbance vs. wavelength) of alcoholic solution of shellac or bleached lac for detection of presence/absence of certain absorption maxima in the UV and visible range.

### 28.3 Apparatus

a) Any spectrophotometer capable of measuring absoibanoe in the range 190/200-700 nm. Equipment with wavelength scan facility with printer would be preferred.

b) Volumetric flask with stopper 100 ml, 10 ml (3 Nos.)

c) Pipettes (graduated) — 2 and 1 ml

d) A weighing balance (accuracy ±0.1 mg), Whatman filter, glass funnel, etc.

e) Quartz cuvettes, 1 cm path length.

### 28.4 Reagent

Ethyl alcohol (absolute)

Price Group 2

**28.5 Procedure**

**28.5.1** Weigh accurately 1 g of finely powdered shellac/bleached lac and transfer to 100-ml volumetric flask. Add about 60 - 70 ml of ethyl alcohol; place the stopper and shake vigorously till the material is dissolved. Add solvent up the mark of the flask. Filter the solution using Whatman No. 1 filter paper preferably under saturated vapour pressure conditions. Discard the first 15 ml of the clear filtrate. The concentration is $10^{-2}$ g/ml.

**28.5.2** *Solution for Absorption Spectrum in the Visible Range*

Transfer 1.5 ml of the filtrate (**28.5.1**) by means of a 2-ml pipette to a 10-ml volumetric flask. Add ethyl alcohol up to the mark. The concentration becomes $1.5\times10^{-3}$ g/ml.

**28.5.3** *Solution for Absorption Spectrum in the UV Range*

Transfer 1 ml of the filtrate (**28.5.1**) using a 1 ml pipette to a 10-ml volumetric flask. Add ethyl alcohol to it up to the mark on the flask. The concentration of the solution is now $10^{-3}$ g/ml. Now again transfer 1 ml of the diluted solution to another 10 ml flask using a 10 ml pipette and make up the volume up to the mark using ethyl alcohol. The concentration is now $10^{-4}$ g/ml.

**28.5.4** *Measurement of Absorption Spectrum in the Visible Range*

Switch on the spectrophotometer. Allow to warm up. Set the wavelength at 700 run. Match the cuvettes with the alcohol to be used for the preparation of the solution. Transfer required quantity of diluted solution outlined under **28.5.2** to one of the cuvettes. Run a wavelength scan between 400-700 nm for absorbance. Study the spectrum.

*Shellac:* Absorption peak at 425 - 430 nm.

*Bleached lac:* The above peak will be absent.

**28.5.5** *Measurement of Absorption Spectrum in the UV Range*

Set the wavelength at 400 nm. Match the cuvettes with the alcohol to be used for the preparation of the solution. Transfer required quantity of diluted solution outlined under **28.5.3** to one of the cuvettes. Run a wavelength scan between 190/200 – 400 nm for absorbance. Study the spectrum.

If the absorption values exceed the upper limit, a solution diluted further may be used for the run.

*Shellac:* Absorption peak at 225, 285 nm and shoulder at 215 nm.

*Bleached lac:* Peaks at 215, 225, 290-300 nm; small peaks at 255, 265 nm.

**28.6 Report**

Presence/absence of peaks and shoulders at the wavelengths specified above are to be mentioned.

**29 DSC ANALYSIS FOR EVALUATING THE QUALITY**

**29.1 General**

The softening (glass transition) and melting temperatures of different forms of lac resin can be determined from their melting profiles (a plot of heat flow against temperature) employing a Differential Scanning Calorimeter. An analysis of the profiles by appropriate software provides accurate and reliable information free from subjectivity. The DSC analysis also provides information on occurrence of resin and wax and purity. The presence of the wax can be determined in shellac, dewaxed lac, bleached lac, etc.

**29.2 Apparatus**

A computer controlled Differential Scanning Calorimeter with provision for different rates of heating and cooling (a minimum of 2°C/min with software for peak position, selection of ranges of dependent and independent variables, determination of glass transition, melting temperatures, partial area, percentage of solid present, purity determination, etc.

The temperature range required for the analysis is 10-120°C.

a) Printer, printing paper for the output from DSC analysis*

b) Aluminium pans with cover for DSC analysis with crimper[41]

c) A weighing balance with accuracy of ±0.01 mg.

**29.3 Procedure**

**29.3.1** Weigh finely powdered 5 - 6mg of lac resin sample, accurately, in the aluminium pan, put the cover and crimp properly.

**29.3.2** Switch on the printer, DSC (precalibrated) and controller. Start purging the sample holder with Ar or $N_2$. Start the compressor/circulator.

*The equipment/material would normally be provided with DSC.

**29.3.3** Place the sample in the crimped pan in the sample holder of DSC.

**29.3.4** Start the software, select the appropriate mem for a temperature profile run. Enter the sample, file name, etc. Enter start and end temperatures, run rate (20°C/min), etc. For different forms of lac resin, the starting temperature should not be more than 20-25°C, as far as possible.

**29.3.5** After attaining the steady state, start run, save the profile after completion of the run.

**29.3.6** Enter into analysis software, select the appropriate portion of the profile, preferably containing the initial and final plateau regions.

**29.3.7** *Glass Transition Temperature*

Open the Tg menu in the software. Position the cursors in the regions. Enter the Tg value. Repeat a few more times by selecting different regions. Selection of Tg is to be made a set of nearly similar values. Care is to be exercised that Tg value is less than the onset temperature if onset is also available in the same menu.

**29.3.8** *Melting Temperature*

Position of the melting peak will be shown on the screen, which can be taken as melting temperature.

**29.3.9** *Purity*

Open the Purity menu. Enter the values of weight of the sample and molecular weight. Select proper regions. Select purity calculation. Repeat the operation for different regions. Select the purity value from a set of near similar values.

**29.4 Examination of Melting Profile**

**29.4.1** *Seedlac, Shellac, Regular Bleached Lac*

Two melting peaks are obtained, at 55-57°C and 72-74°C, due to resin and wax, respectively. The positions may somewhat vary among samples. For bleached lac the resin peak may be found a few degrees higher compared to seedlac and shellac. Since lac is of natural origin, the positions of melting peaks of resin and wax have been found to vary by a few degrees as mentioned above. Reporting may be made about the actual temperatures of peaks observed for resin and wax.

**29.4.2** *Dewaxed Lac, Dewaxed Bleached Lac*

Single melting peak would be found at 55-57°C for dewaxed lac. There will be no peak corresponding to wax. For dewaxed bleached lac the resin peak may be found a few degrees higher compared to seedlac and shellac. No peak for wax (at around 72-74°C) will be observed for dewaxed bleached lac also.

**29.4.3** *Kiri*

Two melting peaks (at 55-57°C and 72-74°C) would be obtained. The height of the resin peak would be lower compared to wax peak. The position of the peaks may be a few degrees higher than seedlac or shellac.

**29.4.4** *Old Seedlac/Shellac*

Polymerized lac does not give any melting peak for the resin. The peak corresponding wax would be found in 72-74°C.

**29.5 Report**

The report should include glass-transition (softening) and melting temperatures (in °C); position of peaks (in °C) and number.

NOTE — For seedlac/shellac, well defined peaks for resin and wax may not be obtained at the first instance in some cases. In order to find well defined peaks, the particular seedlac/shellac sample is to be heated up to 120°C, then a cooling run is to be made preferably at a very stow rate (l – 2°/min) from 120°C to 20/25°C, then the melting run is to be performed as per the method laid down in procedure in 29.3.4 onwards. The profiles will definitely provide better information about the melting peaks.

(CHD 23)

Reprography Unit, BIS, New Delhi, India

# AMENDMENT NO. 1 APRIL 1989
# TO
# IS : 6921 - 1973 METHODS OF SAMPLING AND TEST FOR LAC AND LAC PRODUCTS

( *Page* 16, *clause* 7 ) — Insert the following after 7:

**7.0 General** — The colour index of seed lac and shellac is determined by the method as described in **7.1** and colour of index shellac only is determined by the method as described in **7.2** which may be used as referee method for determination of colour index of shellac.

**7.1 Method**

( *Pages* 16 to 19 *clause* **7.0** to **7.3.3** ) — Renumber the clauses **7.0, 7.1, 7.1.1, 7.1.2, 7.1.3, 7.2, 7.2.1, 7.2.1.1, 7.2.1.2, 7.2.2, 7.3, 7.3.1, 7.3.2,** and **7.3.3** as **7.1.0, 7.1.1, 7.1.1.1, 7.1.1.2, 7.1.1.3, 7.1.2, 7.1.2.1, (a), (b), 7.1,2.2, 7.1.3, 7.1.3.1, 7.1.3.2**, and **7.1.3.3** respectively and delete clause number **7.2.2.1.**

( *Page* 19, *clause* **7.3.3** ) — Insert the following after **7.3.3:**

**7.2 Method 2**

**7.2.0** *Outline of the Method* — This method consists of the measurement optical density of an alcoholic shellac solution ( cone 10 g/l ) at a particular wavelength in the visible range, which after multiplication by 136.9 gives the value of colour index.

**7.2.1** *Apparatus*

**7.2.1.1** *Spectrophotometer* — Any spectrophotometer/Colorimeter ( Grating type ) in the visible range ( 400 — 700 nm ).

**7.2.1.2** *Volumetric flasks* — with stopper ( ground joint ) 100 ml.

**7.2.1.3** *Fipette* — 1 ml.

**7.2.2** *Reagents*

**7.2.2.1** *Alcohol* — Ethyl alcohol ( absolute ) or 95 percent ( by volume ) rectified spirit ( conforming to IS : 323 - 1959* ) or 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959† ) provided that it is colourless.

**7.2.3** *Procedure*

**7.2.3.1** *Preparation of solution* — Weigh accurately 1 g of the prepared

*Specification for rectified spirit ( *revised* ).

†Specification for ordinary denatured spirit ( *revised* ).

sample ( *see* **3.3.2** ) of shellac and transfer the material to the 100 ml volumetric flask. Add 60 - 70 ml of alcohol and shake the flask vigorously as soon as the alcohol is added till the shellac is completely dissolved. Add more solvent, and finally make up the volume up to the mark of the volumetric flask. Filter the solution in an ordinary funnel using medium grade filter paper ( preferably Whatman No. 1 ) and keeping the funnel covered ( best result is obtained if the filtration is done under saturated vapour pressure of the solvent ). Discard the first 15 ml of the clear filtrate.

**7.2.3.2** Transfer one ml of the filtrate by means of a pipette to the 10 ml volumetric flask. Add alcohol to it and make up the volume up to the mark of the flask.

**7.2.3.3** *Measurement of optical density* — Swich on the spectrophotometer/colorimeter. After the warming up period of the instrument, set the wavelength at 425 nm, match the cuvettes with the alcohol used for the preparation of the solution. Transfer a portion of the above diluted solution to one of the cuvettes. Note down the value of the optical density.

**7.2.4** *Calculation*

Colour index = optical density × 136.9

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# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR LAC AND LAC PRODUCTS

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# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR LAC AND LAC PRODUCTS

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 28 February 1973, after the draft finalized by the Lac and Lac Products Sectional Committee had been approved by the Chemical Division Council.

**0.2** A series of Indian Standards for lac and lac products have been published so far. The Committee responsible for these standards felt it desirable to separate the methods of sampling and test from the individual material specifications and publish them together as a self-contained separate Indian Standard.

**0.3** Test sieves prescribed in this standard are based on IS : 460-1962*. Where these sieves are not available, other equivalent standard sieves as judged by the aperture may be used.

**0.4** Acknowledgement is due for the assistance that has been derived from the specifications and publications of the American Society for Testing and Materials; the American Bleached Shellac Manufacturers' Association; the United States Shellac Importers' Association; the British Standards Institution; the Agricultural Marketing Adviser to the Government of India; Messrs Angelo Brothers Ltd, Calcutta; and the Indian Lac Research Institute. Considerable assistance has been derived also from 'A Handbook of Shellac Analysis' by M. Rangaswami and H. K. Sen, issued by the Indian Lac Research Institute. Acknowledgement is due also to the work of the Technical Committee ISO/TC 50 Lac, of the International Organization for Standardization (ISO) for which ISI provided the Secretariat.

**0.5** The methods prescribed in this standard correspond substantially with those published by ISO/TC 50.

**0.6** In reporting the result of a test made in accordance with this standard. if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

## 1. SCOPE

**1.1** This standard prescribes the methods of sampling and test for lac and lac products.

*Specification for test sieves ( *revised* ).

†Rules for rounding off numerical values ( *revised* ).

## 2. TERMINOLOGY

**2.1** For the purposes of this standard, the definitions given in IS : 4908-1968* and the following shall apply.

**2.1.1** *Approved Sample* — The sample agreed upon between the purchaser and the supplier as the standard for colour and general appearance.

## 3. SAMPLING

**3.0** In the case of bleached lac, it is essential that the operations described for drawing, parting and preparation of analysis samples are carried out as expeditiously as possible in order to minimize loss or gain of moisture.

### 3.1 Drawing of Samples

**3.1.1** Only original, unopened packages of the material shall be sampled.

**3.1.2** Not less than 10 percent of the packages, selected at random from each lot, shall be sampled.

**3.1.3** For this purpose a lot shall not exceed 200 packages.

**3.1.4** Unused portions of samples shall be sent to the purchaser on request.

**3.1.5** *Material in a Free Flowing Condition* — Samples shall be taken from different places in each package by means of a suitable tryer so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. The material shall then be thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters mixed. One half of the material shall be labelled as the 'original observation sample' and shall, as necessary, be further sub-divided by the normal process of quartering to form a number of original observation samples ( *see* **3.1.8** ). The other half of the material shall be treated as described under **3.2.1** to form the 'analysis sample'.

**3.1.6** *Blocky or Matted Material* — Samples shall be taken from different places in each package by chipping or other suitable means so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. The material shall then be halved by suitable means. One half of the material shall be labelled as the 'original observation sample' and shall, as necessary, be further subdivided to form a number of original observation samples ( *see* **3.1.8** ). The other half of the material shall be roughly ground so as to pass 6.3-mm ( PS ) IS Sieve and shall then be treated as described under **3.2.1** to form the 'analysis sample'.

**3.1.7** *Bleached Lac in Hanks, Bars or Flats* — Two hanks, bars or flats shall be drawn from different places in each package and a large piece shall be broken

*Glossary of terms used in lac industry.

from each by suitable means so as to yield a total of 5 kg of material consisting of approximately equal portions from each hank, bar or flat. The composite sample shall be quickly crushed to lumps of about 25 mm cube. The material shall then be halved by suitable means. One half of the material shall be labelled as the 'original observation sample' and shall, as necessary, be sub-divided by the normal 'process of quartering ( without crushing ) to form a number of 'original observation samples' ( *see* **3.1.8** ). The remaining half of the material shall be roughly ground so as to pass 6.3-mm ( PS ) IS Sieve and shall then be treated as described uner **3.2.1** to form the 'analysis sample'.

**3.1.8** An original observation sample shall serve for the determination of volatile matter (moisture) and particle size, if these requirements are among the mandatory requirements or are agreed upon between the purchaser and the supplier. The original observation samples shall be placed in air-tight containers, scaled and labelled as 'original observation sample [ sample for the determination of volatile matter (moisture) and particle size ]'.

### 3.2 Parting of Samples

**3.2.0** In the case of bleached lac, if the material at any time during the following operations shows signs of surface moisture, it shall be air-dried at room temperature before further mixing and grinding.

**3.2.1** The material for the 'analysis sample' as obtained under **3.1.5** or **3.1.6** or **3.1.7** shall be mixed thoroughly, and heaped and quartered along two diameters which intersect at right angles. Two opposite quarters shall be mixed and ground to pass entirely through 2.00-mm IS Sieve. The material shall then be thoroughly mixed and twice halved by quartering so as to yield 4 samples of approximately 250 g each. These 4 samples shall be placed in air-tight containers, sealed and labelled as 'sample for analysis' and sent to the interested parties.

**3.2.2** The date of sampling, the number of packages sampled, the condition of the packages and contents, and the name and code number of the supplier shall be given on a label attached to each sample.

### 3.3 Preparation of Analysis Samples for Testing

**3.3.1** The samples for analysis shall be ground to pass entirely through sieves of nominal aperture size as given below:

| | |
|---|---|
| Seedlac | 425-micron IS Sieve |
| Shellac | 425-micron IS Sieve<br>*or*<br>500-micron IS Sieve<br>*or*<br>710-micron IS Sieve |
| Bleached lac | 425-micron IS Sieve |

**3.3.2** *For Seedlac and Shellac* — The finely ground material shall be mixed thoroughly and divided into the requisite number of samples for testing, in accordance with the requirements of the material specification. These samples shall be placed in air-tight containers, sealed and labelled as 'prepared samples'.

**3.3.3** *For Bleached Lac* — The finely ground material shall be mixed thoroughly and placed in an air-tight container and labelled 'unconditioned sample for analysis'. Before this material is used for any analytical work, it shall be brought to less than 6 percent volatile matter ( moisture ) content by exposing it to the atmosphere for at least 24 hours at room temperature and then desiccating overnight over fused calcium chloride. The material shall then be known as 'prepared sample'.

**3.3.4** The original observation sample, which is to be used for the determination of volatile matter (moisture) and particle size, when these requirements are agreed upon between the purchaser and the supplier, shall be treated as prescribed below:

*Seedlac* — It shall be ground to pass entirely through 850-micron IS Sieve.

*Shellac* — It shall be ground to pass entirely through 425-micron IS Sieve *or* 500-micron IS Sieve *or* 710-micron IS Sieve.

*Bleached lac* — It shall be treated according to the method laid down in **5.2.**

## 4. QUALITY OF REAGENTS

**4.1** Unless specified otherwise, pure chemicals and distilled water *( see* IS : 1070-1960* ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the remits of analysis.

## 5. DETERMINATION OF VOLATILE MATTER ( MOISTURE )

### 5.1 For Seedlac and Shellac

**5.1.0** *Outline of the Method* — The volatile matter ( moisture ) content is determined by heating a weighed specimen of the 'original observation sample' at 41 ± 1°C for 4 hours and then keeping it over concentrated sulphuric acid *in vacuo* for 18 hours.

**5.1.1** *Procedure*

**5.1.1.1** For this test, use the 'original observation sample' ground to specified size ( *see* **3.3.4** ). Weigh a clean, dry, flat-bottomed dish of about

*Specification for water, distilled quality ( *revised* ).

75 mm diameter provided with a ground glass cover. Transfer approximately 2 g of the powdered sample to the dish, cover it with the ground glass cover and weigh it again. The difference gives the mass of the sample taken.

**5.1.1.2** Keep the dish with the sample, without covering it, in a well-ventilated oven maintained at 41 ± 1°C for 4 hours. At the end of this period, transfer the dish and cover to a vacuum desiccator containing concentrated sulphuric acid. Immediately evacuate the desiccator and keep the sample uncovered *in vacuo* for 18 hours. Remove the dish, cover it with the ground glass cover and immediately weigh. Express the loss in mass as a percentage of the mass of the sample taken.

**5.1.2** *Calculation*

$$\text{Volatile matter ( moisture ), percent by mass} = \frac{100\,(M - m)}{M}$$

where

$M$ = mass in g of sample taken, and

$m$ = mass in g of sample after drying.

## 5.2 For Bleached Lac

**5.2.0** *Outline of the Method*

**5.2.0.1** The volatile matter ( moisture ) content of bleached lac is determined in two stages, the first stage being by drying a weighed specimen of the 'original observation sample' by keeping over concentrated sulphuric acid *in vacuo* for 12 to 24 hours. In the case of bone-dry bleached lac, this first stage may be omitted.

**5.2.0.2** For the second stage, grind the partially dried material thus obtained to the specified size and further dry a portion by heating in a well-ventilated oven at 41 ± 1°C for 18 hours.

**5.2.1** *Procedure*

**5.2.1.1** Use a portion of the 'original observation sample' ( *see* **3.3.4** ) and crush, if necessary, into granules using a heavy pestle and mortar, keeping the latter covered as far as possible during the process. Weigh a clean, dry, flat-bottomed dish of about 100 mm diameter provided with a glass cover. Transfer approximately 10 g of the ground sample to the dish as rapidly as possible, cover it with the glass cover and reweigh. The difference gives the mass of sample taken.

**5.2.1.2** Transfer the dish and contents to a vacuum desiccator containing concentrated sulphuric acid and remove the cover of the dish. Evacuate the desiccator and keep the sample uncovered *in vacuo* for not less than 12 hours and not more than 24 hours. Remove the dish, replace the cover

and weigh. The difference between this and the mass of the dish is the mass of the partially dried sample. Grind the partially dried sample thus obtained until it passes through 400-micron IS Sieve.

**5.2.1.3** Weigh approximately 2 g of the ground material to an accuracy of 1 mg into a covered tared dish of the type described under **5.2.1.1** and transfer to a well-ventilated oven maintained at 41 ± 1°C for 18 hours, the cover of the dish being removed during the drying process. At the conclusion of the heating period, replace the cover and transfer the covered dish to a desiccator; weigh when cool. This mass minus the mass of the dish is the mass of the completely dried ground sample.

**5.2.2** *Calculation*

**5.2.2.1** The percentage of volatile matter ( moisture ) in the original observation sample is given by the following formula:

$$\text{Volatile matter ( moisture ), percent by mass} = 100\left[1 - \frac{M_4 M_2}{M_3 M_1}\right]$$

where

$M_1$ = mass in g of crushed original sample taken for drying *in vacuo;*

$M_2$ = mass in g of partially dried sample;

$M_3$ = mass in g of partially dried, ground sample taken for final oven drying; and

$M_4$ = mass in g of completely dried, ground sample.

**5.2.2.2** If the first drying stage ( *see* **5.2.1.2** ) be omitted, then the percentage of volatile matter ( moisture ) in the 'original observation sample' is given by the formula:

$$\text{Volatile matter ( moisture ), percent by mass} = 100\left[1 - \frac{M_2}{M_1}\right]$$

where

$M_1$ = mass in g of sample taken for oven drying; and

$M_2$ = mass in g of completely dried, ground sample.

## 6. DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

**6.0 General** — The matter insoluble in hot alcohol is determined by extracting a known mass of the sample with 95 percent ( by volume ) ethyl alcohol and determining the percentage of the undissolved residue by either of the two methods ( as may be agreed to ) described under **6.1** and **6.2**.

## 64 Method I

**6.1.1** *Apparatus* — The apparatus shall consist of the following.

**6.1.1.1** *Condenser* — all glass, of the type and dimensions shown in Fig. 1, with the tip cut at an angle of 45 degrees. It shall have two holes at the tip to fasten the siphon tube.

**6.1.1.2** *Siphon tube* — of glass, of the type and dimensions shown in Fig. 1. The siphon tube shall have 2 holes near the top for a wire to be fastened to the condenser tip, leaving about 6 mm space between the top of the tube and the condenser tip.

**6.1.1.3** *Flask* — heat-resistant, wide mouth, conical type, preferably of borosilicate glass, 176 ± 3 mm in height and 48 ± 2 mm in inside diameter at the top. The flask shall have a tight fitting cork, 25 mm in depth and bored to fit the stem of the condenser. The bottom of the cork shall be just above the holes for the wire in the condenser. To support the flask, a suitable ring support with iron clamp and nichrome or iron gauze shall be used. The gauze shall not have an asbestos covering.

**6.1.1.4** *Filter tube* — a carbon filter tube of the type and dimensions shown in Fig. 1, with a light spiral spring at the bottom to hold up the extraction cartridge. The stem of the filter tube shall be fitted with a rubber stopper and firmly held in a hot water bath.

**6.1.1.5** *Extraction cartridge* — fat-free paper extraction cartridge* 26 ± 1 mm in diameter and 60 ± 1 mm in height.

**6.1.1.6** *Weighing bottle* — glass-stoppered, 80 ± 1 mm in height and 40 ± 1 mm in diameter.

**6.1.1.7** *Hot water bath* — made of about 0.9 mm thick copper, having the dimensions given in Fig. 2. The cover shall have a flanged hole 57 ± 1 mm in diameter for a 200-ml beaker, and also a hole 35 ± 1 mm in diameter through which the top of the filter tube projects. Directly below this hole, in the bottom of the bath, shall be a flanged hole 25 ± 1 mm in diameter to hold the rubber stopper through which the stem of the filter tube extends, to discharge into the bottle or flask. The hot water bath shall be mounted on a low tripod or stand.

**6.1.1.8** *Gas burner* — low form, adjustable, Bunsen type, carrying a draught shield. Any other suitable heating device may also be employed.

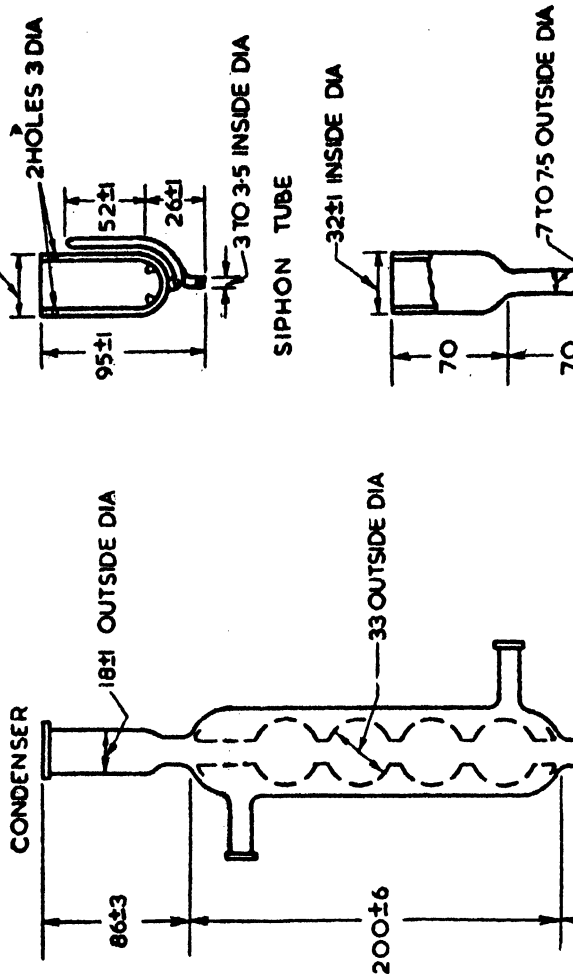
**6.1.2** *Reagent*

**6.1.2.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959† ); or 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959‡ )

*Schleicher and Schull No. 603 or equivalent is suitable.

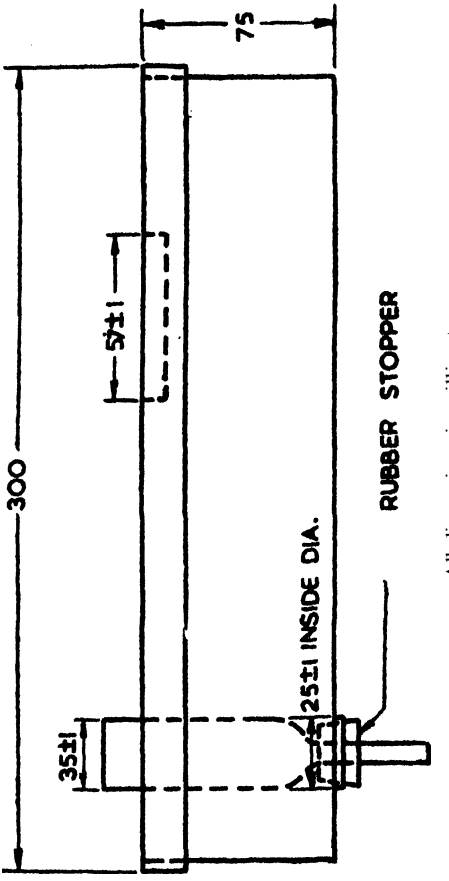
†Specification for rectified spirit ( *revised* ).

‡Specification for ordinary denatured spirit ( *revised* ).




All dimensions in millimetres.

FIG. 1 EXTRACTION APPARATUS FOR DETERMINING MATTER INSOLUBLE IN HOT ALCOHOL, METHOD I




All dimensions in millimetres.

FIG. 2 HOT WATER BATH FOR DETERMINING MATTER INSOLUBLE IN HOT ALCOHOL, METHOD I

**6.1.3** *Preparation of Extraction Cartridge* — Place 125 ml of the alcohol in the flask and a cartridge in the siphon tube. Introduce the siphon tube into the flask and connect it to the condenser, making sure that there is an ample flow of cold water through the condenser. Adjust the flame of the burner so as to give a cycle of filling and emptying in the siphon tube every 2 minutes, and extract for 30 minutes. Dry the cartridge in an oven at a temperature not exceeding 105°C. At the end of 3 hours, weigh it in a tared weighing bottle which has been kept in a desiccator over sulphuric acid, lifting the stopper of the bottle momentarily before weighing. Continue drying, and weigh as before after each hour of drying, until the loss in mass between successive weighings does not exceed 2 mg.

**6.1.3.1** For referee tests new cartridges only shall be used. A number of cartridges may be extracted, dried, weighed and kept in weighing bottles in a desiccator until needed for use.

**6.1.4** *Procedure*

**6.1.4.1** Before analysis, thoroughly mix the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) by rolling on paper at least 10 times, to ensure uniformity of the analytical sample. Weigh, directly from the rolling sheet, 4.5 to 5.5 g of the sample to an accuracy of 0.01 g, place in a 200-ml, tall, lipped beaker, add 125 ml of alcohol, stir with a glass rod, cover with a watch glass, and place in the hot water bath ( *see* Fig. 2 ). Boil the solution vigorously for 30 minutes to ensure complete solution of the sample and dispersion of wax. Keep the volume of alcohol constant.

**6.1.4.2** Meanwhile place an extracted and weighed cartridge in the filter tube. Maintain the hot water around the tube at not less than 90°C. Wet the cartridge with hot alcohol, and decant the boiling solution into the heated cartridge until the beaker is nearly empty.

**6.1.4.3** Wash the remaining solution and the insoluble matter into the cartridge, using a 'policeman', if necessary, with successive portions of hot alcohol contained in a wash bottle kept ho on the water bath. Finally, wash the cartridge from the top downwards with a fine stream of hot alcohol. A complete washing and transfer from the original beaker will require at least 75 ml of hot alcohol.

**6.1.4.4** Transfer the cartridge containing the insoluble matter to the extraction apparatus, place 125 ml of alcohol in the extraction flask and connect up the apparatus. Start the water flowing through the condenser, making sure that there is an adequate supply for efficient condensation. Light the burner and time the extraction from the first emptying of the siphon, running the extraction for exactly one hour. Adjust the Bunsen burner so that a complete filling and emptying of the siphon tube takes place every 2 minutes, as determined by a stop watch, or preferably by a good two-minute sand glass, one for each extraction apparatus.

NOTE 1 — In this way exactly 30 cycles per hour are accomplished. If this cycle is not meticulously maintained, neither check results on duplicate samples in the same

laboratory, nor concordant figures from one laboratory to another can be obtained, even when working on the same sample. It is also necessary to guard the apparatus from draughts while in operation; otherwise the proper cycle rate cannot be maintained.

NOTE 2 — Occasionally, samples are encountered which do not yield the required number of 30 siphonings per hour, due to slow filtration. In these instances, continue the extraction until 30 siphonings have been accomplished or repeat the test with a 2 g sample, and report the sample as abnormal or slow filtering.

**6.1.4.5** Remove the cartridge, drain in an upright position on filter paper and dry in an oven at 100 ± 2°C. After drying for 2 hours, place it in the weighing bottle, cool in a desiccator over sulphuric acid, and weigh, removing the stopper momentarily just before weighing. Repeat drying and weighing as before, after each hour of drying, until the loss in mass between successive weighings does not exceed 2 mg. From the mass of the residue and the mass of the sample, calculate the percentage of insoluble matter.

**6.1.5** *Calculation*

$$\text{Matter insoluble in hot alcohol, percent by mass} = \frac{100 \times M_1}{M_2}$$

where

$M_1$ = mass in g of residue, and

$M_2$ = mass in g of sample taken.

NOTE — For calculating results on the basis of moisture-free sample, multiply the value obtained by:

$$\frac{100}{100 - \text{moisture percent}}$$

where moisture percent has been obtained from 5.

## 6.2 Method II

**6.2.1** *Apparatus* — The apparatus shall consist of the following.

**6.2.1.1** *Siphon tube* — of glass, of the Knoefler type having minimum internal dimensions of 52 mm height and 32 mm diameter, resting in an adaptor tube in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent ( *see* Fig. 3 ).

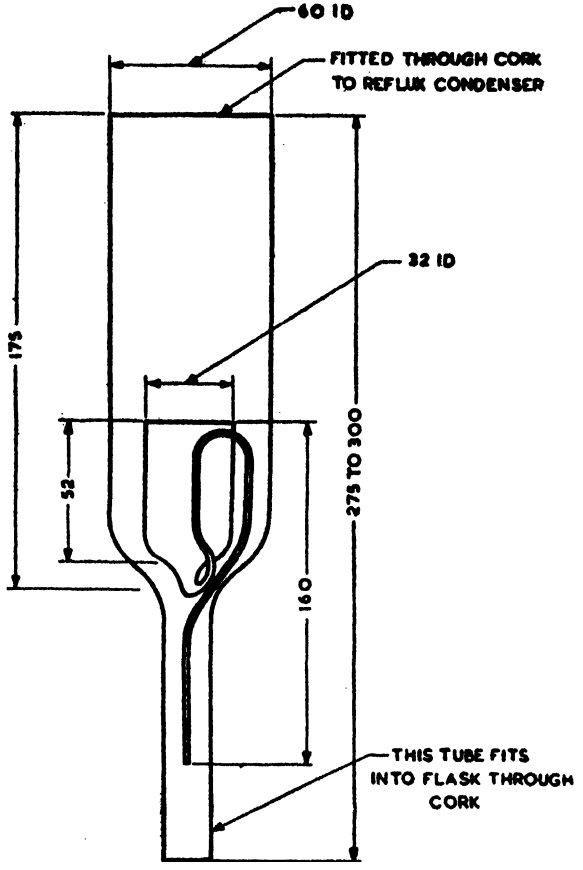
**6.2.1.2** *Condenser* — of any convenient pattern.

**6.2.1.3** *Flask* — of any convenient size.

**6.2.1.4** *Filter paper* — 12.5 cm diameter, medium grade ( Whatman No. 1 or equivalent ).

**6.2.1.5** *Weighing bottles* — of glass, height 80 ± 1 mm, and diameter 40 ± 1 mm, with ground glass stoppers.

NOTE — The type of extraction apparatus used is not critical, provided that it is of such a design as to ensure a continuous series of extractions at approximately the boiling temperature of the solvent. If preferred, the apparatus described in **6.1.1** could be satisfactorily used.




All dimensions in millimetres.

FIG. 3 SIPHON TUBE AND ADAPTOR

**6.2.2** *Assembly of Apparatus* — The siphon tube, adaptor, condenser and flask shall be assembled with the aid of corks or ground glass joints so that the solvent can be kept boiling in the flask and its vapour passed upwards by way of the adaptor to the condenser. The refluxing solvent shall run from the condenser into the cup of the siphon tube.

**6.2.3** *Reagent*

**6.2.3.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959* ); or 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959† ).

*Specification for rectified spirit ( *revised* ).

†Specification for ordinary denatured spirit ( *revised* ).

**6.2.4** *Procedure*

**6.2.4.1** Fold a filter paper so that it forms a completely closed envelope as illustrated in Fig. 4. Mark this paper *'S'* for sample; wrap it closely in a second filter paper marked *'C'* for counterpoise. Separate the filter papers and dry in an oven at 100 ± 2°C for 30 minutes. Rapidly transfer to weighing bottles which have been kept in a desiccator over concentrated sulphuric acid. Place each bottle with its contents back in the desiccator for 20 minutes, then weigh by counterpoise, preferably using a rapid weighing balance of the aperiodic type.

**6.2.4.2** Weigh 4.5 to 5.5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.01 g and place in the filter paper envelope *S* and fold in the original folds taking care not to leave any channel through which finely divided material might afterwards escape. Again enclose in paper *C* and secure with thread. Place the resulting envelope in a 100-ml beaker and cover it with alcohol. Allow to stand overnight at room temperature. Transfer the envelope to the cup of the siphon tube and extract continuously with hot alcohol for 4 hours. Keep the envelope wholly below the surface of the alcohol when the cup is full. Maintain a rapid rate of extraction throughout, though the exact time taken for the cycle of filling and emptying the cup of the siphon tube is not critical.

NOTE — Double folds on the three sides, as illustrated in (3) of Fig. 4, are recommended to ensure against inadvertent escape of finely divided material.

**6.2.4.3** At the end of the specified time, remove the paper envelope, allow to drain, separate the two papers, dry each on a glass plate in the steam oven and then for 5 hours in a thermostatically controlled oven at 100 ± 2°C. Place the papers rapidly in their respective weighing bottles, allow to stand in the desiccator for 20 minutes and again weigh by counterpoise after momentarily removing and replacing the stoppers in the usual manner. Dry the papers for a further period of one hour at 100 ± 2°C and weigh again; if there is a loss in mass in excess of 2 mg, repeat the processes of drying and weighing until the difference between successive weighings is less than 2 mg. Use the lowest mass in the calculation.

**6.2.5** *Calculation* — as given in **6.1.5.**

## 7. DETERMINATION OF COLOUR INDEX OF SEEDLAC AND SHELLAC

**7.0 Outline of the Method** — The colour index is determined by comparing the colour of a standard solution of iodine with a solution of the sample in ethyl alcohol by diluting the sample solution progressively with alcohol until a close match is obtained.

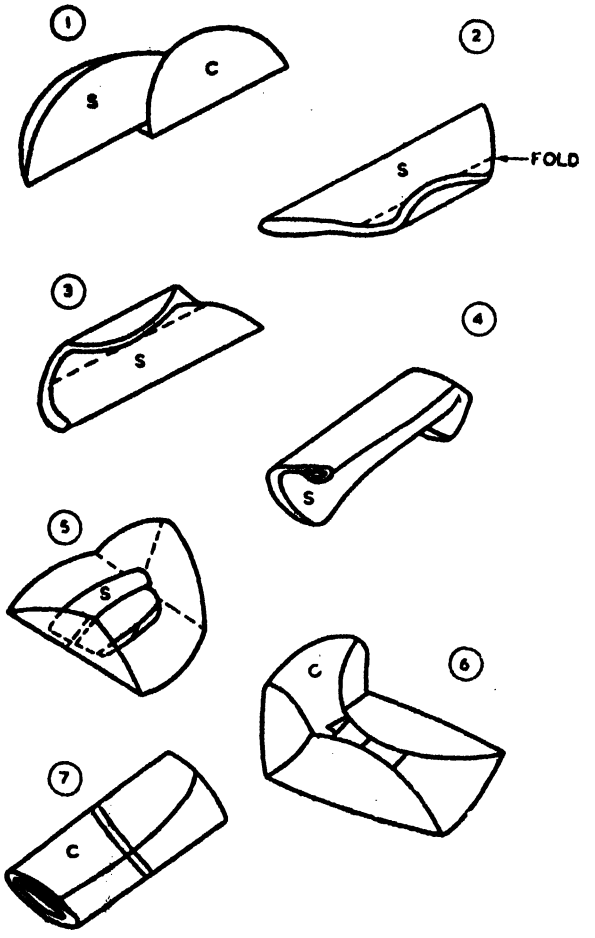



FIG. 4 FOLDING OF FILTER PAPER

## 7.1 Reagents

**7.1.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959* ); or 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959† ); provided that it is colourless.

*Specification for rectified spirit ( *revised* ).

†Specification for ordinary denatured spirit ( *revised* ).

**7.1.2** *Standard Iodine Solution,* 0.005 N — Prepare the solution by introducing 5 ml of 0.1 N iodine solution (in potassium iodide solution), with a burette, into a measuring flask and making up to 100 ml with water. This solution corresponds to colour index 5. Shake the solution before use. Use this solution for hand-made shellac, machine-made shellacs which have not been decolourized and for seedlac.

**7.1.3** *Standard Iodine Solution,* 0.001 N *(for Decolourized Shellacs )* — Prepare freshly by appropriate dilution of a 0.1 N solution of iodine in potassium iodide with water. The colour of the 0.001 N solution corresponds to colour index 1.

## 7.2 Procedure

**7.2.1** *Preparation of Solution*

**7.2.1.1** *Seedlac* — Add 100 ml of alcohol to 100 g of the 'prepared sample' ( *see* **3.3.2** ) contained in a stoppered flask. Shake vigorously as soon as alcohol is added, to prevent coalescence of lac particles, and then intermittently over a period of 4 hours. Allow to stand for 16 to 24 hours at 27 ± 2°C, shake again, and allow to settle at 27 ± 2°C for 2 hours. Filter the solution in an ordinary funnel using medium grade filter paper and keeping the funnel covered. Discard the first 15 ml or more of the filtrate and then collect 5 ml of the clear filtrate for the test.

**7.2.1.2** *Shellac* — Dissolve 10.0 g of the 'prepared sample' ( *see* **3.3.2** ) in 100 ml of the alcohol by stirring for 30 minutes at 27 ± 2°C. Filter the solution in an ordinary funnel using a medium grade filter paper. Discard the first 15 ml or more of the filtrate and then collect 5 ml of the clear filtrate tor the test.

**7.2.2** Transfer 5 ml of the filtered sample solution to a thin-walled test-tube measuring 200 × 13 mm by means of a pipette. Take 5 ml of the appropriate standard iodine solution in another test-tube similar in every respect for matching. Compare the colour of the two solutions holding the test-tubes against light with a piece of moistened filter paper or opal glass interposed in between the light source and the test-tubes. Add alcohol from a burette to the sample solution with shaking until the colour is the same as that of the standard solution. Note the volume of alcohol added. Alternatively, in the case of decolourized shellac, if the sample solution is lighter, dilute the standard iodine solution with water contained in a second burette until a match is obtained and note the volume added.

**7.2.2.1** It will be found advantageous to use a standard type of light source and a viewing cabinet to cut off extraneous light.

## 7.3 Calculation

**7.3.1** *For Seedlac and Non-decolourized Shellac* — The volume in millilitres of alcohol added plus five, or the total volume in millilitres of the sample solution after dilution, is the colour index of the sample.

**7.3.2** *For Decolourized Shellac*

$$\text{Colour index} = \frac{A + 5}{V + 5}$$

where

$A$ = volume of alcohol in ml added to the sample solution, and

$V$ = volume of water in ml added to the standard iodine solution.

**7.3.3** The accuracy of this test, including the personal error of different analysts, is about 5 percent.

## 8. TEST FOR COLOUR OF BLEACHED LAC

**8.0 General** — Two methods for the determination of colour are described. One of these is based on the visual comparison of solutions or films prepared from the material and the approved sample; the other is based on the determination of colour index of the material and the approved sample by visual comparison of intensity of colour of a 10 percent ( *m/v* ) solution of the material in 95 percent ( by volume ) alcohol with a standard solution of iodine.

### 8.1 Method I

**8.1.0** *Outline of the Method* — Bleached lac is dissolved in 95 percent ( by volume ) ethyl alcohol and the colour examined visually against similarly treated approved sample either in the solution form or after forming films.

**8.1.1** *Procedure* — Treat a weighed portion of bleached lac with twice its mass of cold 95 percent ( by volume ) rectified spirit, conforming to IS : 323-1959*, shaking at intervals until the lac is entirely dissolved. Compare its colour with the colour of a similar solution of the approved sample ( *see* Note ) by any of the following methods, as agreed to between the purchaser and the supplier.

NOTE — It should be noted that the insoluble solids content of the approved sample shall be the same as that of the bleached lac sample under test.

**8.1.1.1** *Comparison of solutions* — Shake both solutions well and then place equal portions of each in separate clear-glass tubes of the same diameter. Place the tubes together and compare the colour visually.

**8.1.1.2** *Comparison of films* — Shake the solutions well and then allow them to stand for 30 minutes. Flow approximately equal portions of the two bleached lac solutions on separate milk glass plates or porcelain plates and allow the films to dry in a vertical position. Make the colour comparison Upon dried films visually.

*Specification for rectified spirit ( *revised* ).

## 8.2 Method II

**8.2.0** *Outline of the Method* — The colour is determined by comparing the colour of a 0.001 N solution of iodine ( in potassium iodide solution ) with a solution of bleached lac in ethyl alcohol by diluting the darker solution progressively until a close visual match is obtained, and then repeating the test with a solution of the approved sample.

**8.2.1** *Reagents*

**8.2.1.1** *Alcohol* — same as in **7.1.1.**

**8.2.1.2** *Standard iodine solution,* 0.001 N — same as in **7.1.3.**

**8.2.2** *Procedure*

**8.2.2.1** *Preparation of solution* — Dissolve 10.0 g of the 'prepared sample' ( *see* **3.3.3** ) in 100 ml of alcohol by shaking for 30 minutes at 27 ± 2°C. Filter the solution in an ordinary funnel using a medium grade filter paper. Discard the first 15 ml or more of the filtrate and then collect 5 ml of the clear filtrate for the test.

**8.2.2.2** Transfer 5 ml of the filtered solution to a thin-walled test-tube measuring 200 × 13 mm by means of a pipette. Take an adequate volume of the standard iodine solution in another test-tube, similar in every respect, for matching. Compare the colour of the two solutions holding the test-tubes against light with a piece of moistened filter paper or opal glass interposed in between the light source and the test-tubes. Add alcohol from a burette to the darker solution with shaking until the colour of both the solutions is the same. Note the volume of alcohol added. Repeat using the approved sample in place of the sample under test.

NOTE — It will be found advantageous to use a standard type of light source and a viewing cabinet to cut off extraneous light.

**8.2.3** *Reporting* — It shall be reported whether the solution prepared from the sample under test is found to be lighter than that prepared from the approved sample, or equal in darkness, or darker.

## 9. DETECTION OF ROSIN ( HALPHEN-HICKS METHOD )

### 9.1 Reagents

**9.1.1** *Ethyl Alcohol — absolute* ( conforming to IS : 321-1964* ).

**9.1.2** *Acetic Acid* — glacial.

**9.1.3** *Petroleum Ether* — boiling point below 80°C.

**9.1.4** *Solution A* — one part of phenol by volume dissolved in 2 parts by volume of carbon tetrachloride.

*Specification for absolute alcohol ( *revised* ).

**9.1.5** *Solution B* — one part by volume of bromine dissolved in 4 parts by volume of carbon tetrachloride.

### 9.2 Procedure

**9.2.1** Place about 2 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) in a 250-ml conical flask, add 10 ml of ethyl alcohol or acetic acid, and shake until solution of the resinous material is complete. Then add slowly and with continuous agitation 50 ml of petroleum ether. After the addition of petroleum ether, add 50 ml of water in exactly the same manner, transfer to a small separating funnel, and allow it to stand until the petroleum ether separates. Draw off the water layer, wash the petroleum ether layer once with water, and then filter the petroleum ether extract through a dry filter paper into a round-bottom evaporating dish. Evaporate to dryness on a steam bath and test the residue as follows.

**9.2.2** Add 1 to 2 ml of Solution A to the residue and pour this mixture into a cavity of an ordinary porcelain colour-reaction plate until it just fills the depression. Immediately fill an adjacent cavity with Solution B. Cover the plate with an inverted watch glass and note the colour, if any, produced in Solution A or its creeping edge by the action of bromine vapours from Solution B.

**9.2.3** A purple or deep indigo blue colour is an indication of the presence of rosin.

## 10. DETECTION OF COPALS IN BLEACHED LAC

### 10.1 Reagents

**10.1.1** *Denatured Alcohol* — Add 5 parts by volume of methyl alcohol ( 99 percent ) to 100 parts by volume of 95 percent rectified spirit ( conforming to IS : 323-1959* ).

**10.1.2** *Methyl Alcohol* — 99 percent.

**10.2 Procedure** — Dissolve a weighed portion ( 10 to 20 g ) of the 'prepared sample' ( *see* **3.3.3** ) in twice its mass of denatured alcohol and filter the solution through filter paper. Transfer approximately 10 ml of the clear filtrate to a large test-tube, 150 × 20 mm. Nearly fill the test-tube with methyl alcohol, stopper the tube and mix the contents thoroughly.

**10.2.1** Bleached lac free from copals should remain clear. Immediate formation of a precipitate indicates the presence of copals.

## 11. DETERMINATION OF WAX

**11.0** Two methods for the determination of wax, Method I for seedlac, shellac containing wax, and regular bleached lac, and Method II for dewaxed shellac and refined bleached lac, are described below.

*Specification for rectified spirit ( *revised* ).

## 11.1 Method I

**11.1.0** *Outline of the Method* — A specified quantity of the sample is dissolved in a hot solution of sodium carbonate, the wax separated by filtering suitably, extracted by means of chloroform and weighed after drying.

**11.1.1** *Reagents*

**11.1.1.1** *Sodium carbonate* — anhydrous, analytical reagent.

**11.1.1.2** *Chloroform* — redistilled, free from non-volatile residue.

**11.1.1.3** *Filter aid* — a suitable filter aid*, previously extracted with chloroform and dried before use.

**11.1.2** *Procedure*

**11.1.2.1** Weigh 9.5 to 10.5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.01 g and dissolve in 150 ml of hot water containing 2.5 g of sodium carbonate in a 200-ml tall beaker. Immerse the beaker in a steam or boiling water bath, and stir until the sample is in solution. Then cover with a watch glass and allow it to remain in the bath for 2 to 3 hours more, without agitation. Remove the beaker from the bath, and place it in cold water. The wax will now come to the top and either solidify as a layer or float as small, hard particles, according to the amount of wax present in the sample. Either filter this solution through a 12.5-cm double acid washed retentive, low ash filter paper ( Whatman No. 40 or equivalent ) by gravity, or use a Buchner funnel with suction. In the latter case, it is necessary to embed the filter paper in the Buchner with filter aid, by mixing 1 g of the filter aid with water and pouring this mixture on the paper with the suction on. Filtration by this method is also further aided by stirring 0.5 g of the filter aid into the sample solution before starting the filtration.

**11.1.2.2** In case the filtration is done only under gravity, after the filtration is completed and all soluble sample washed out of the paper with water, remove the paper from the funnel and without further folding it, set it in the beaker, resting against the stirring rod so that the edge of the paper remains level with the top edge of the beaker. Keep the beaker containing the paper in an oven maintained at 40 ± 2°C for several hours to remove most of the water. Next, remove the paper from the beaker, wrap carefully in a larger piece of clean fat-free filter paper, bind with the fine wire and place it in a 26 × 60 mm fat-free paper extraction cartridge† which has been previously extracted with hot chloroform. Put the cartridge containing the wax and paper into a suitable continuous extraction apparatus such as the standard hot alcohol insoluble apparatus ( **6.1.1** ) and pour into the beaker, which

*An example of a suitable filter aid is a diatomaceous material sold under the name of 'Filter-cel'.

†Schleicher and Schull No. 603 or equivalent is suitable.

previously contained the filter paper and wax, a portion of the chloroform to be used for the extraction. Bring the solvent to boil, and pour it through the extraction cartridge, collecting it in the extraction flask to be used. Repeat this operation twice more so as to remove the whole of the residual wax from the beaker. Then connect up the apparatus and extract for at least 3 hours. Distil off most of the solvent, transfer the residue to a tared glass basin, wash the extraction flask three times with small lots of 5 ml of chloroform and pour into the basin. Evaporate to dryness, and then dry the wax at 100 ± 2°C until the loss does not exceed 0.000 2 g in half-hourly periods of heating. Use the lowest mass in calculation.

**11.1.2.3** If the Buchner funnel is used, after the filtration has been completed and the paper has been well washed with water to take out all soluble matter, the vacuum is left on for a few minutes so as to suck out as much water as possible. It will then be possible to insert a thin spatula under the edge of the paper and remove it from the funnel, without leaving more than traces of the filter aid adhering to the funnel walls. Remove such traces by wiping with bits of alcohol-moistened paper, combine these with the main paper, and wrap the whole, while still damp, in a large piece of filter paper, and bind firmly with fine wire. Dry this at a temperature of 40 ± 2°C. When dry, place it in a 26 × 60 mm fat-free paper extraction cartridge* which has been previously extracted with chloroform. Transfer the cartridge and wax to any suitable continuous extraction apparatus, such as the standard apparatus for hot alcohol insoluble matter ( **6.1.1** ), and extract for 2 hours with chloroform. Distil off most of the solvent, transfer the residue to a tared glass basin, wash the extraction flask three times with small lots of 5 ml of chloroform and pour into the basin. Evaporate to dryness, and then dry the wax at 100 ± 2°C until the loss does not exceed 0.000 2 g in half-hourly periods of heating. Use the lowest mass in calculation.

**11.1.3** *Calculation*

$$\text{Wax, percent by mass} = \frac{100\,m}{M}$$

where

$m$ = mass in g of wax, and

$M$ = mass in g of sample taken.

NOTE — For calculating results on moisture-free basis, *see* Note under **6.1.5.**

## 11.2 Method II

**11.2.0** *Outline of the Method* — A specified quantity of the sample is dissolved in alcohol, and the wax separated out at 0°C with the use of a filter aid and oxalic acid; the wax is subsequently extracted from the filter aid by means of chloroform and weighed after drying.

*Schleicher and Schull No. 603 or equivalent is suitable.

**11.2.1** *Reagents*

**11.2.1.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959* ).

**11.2.1.2** *Oxalic acid* — dihydrate.

**11.2.1.3** *Chloroform* — redistilled, free from non-volatile residue.

**11.2.1.4** *Filter aid* — a suitable filter aid†, previously extracted with chloroform and dried before use.

**11.2.2** *Procedure* — Weigh 49 to 51 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.1 g and dissolve in 250 ml of alcohol, add about 1 g of oxalic acid and stir until all is dissolved. Then add about 0.5 g of the filter aid and allow to flocculate and settle overnight at 40 ± 1°C. Now cool to 0°C and keep at this temperature for at least one hour. Pour the clear liquid through a Gooch crucible prepared with an asbestos mat over which a thin layer of filter aid has been applied. Wash the sediment of wax and the filter aid from the beaker into the crucible with cold alcohol at 0°C. Dry at 41 ± 1°C. Remove the asbestos mat, wipe out the crucible with small pieces of alcohol-moistened paper and wrap the mat and the wipings carefully in a large piece of fat-free filter paper. Bind securely with fine wire and transfer to a previously extracted 26 × 60 mm fat-free paper extraction cartridge and extract in a suitable continuous extraction apparatus, such as the hot alcohol insoluble apparatus ( **6.1.1** ), with chloroform for 2 hours. Distil off most of the solvent, transfer the residue to a tared glass basin, wash the extraction flask three times with small lots of 5 ml of chloroform and pour into the basin. Evaporate to dryness, and then dry the wax at 100 ± 2°C until the loss does not exceed 0.000 2 g in half-hourly periods of heating. Use the lowest mass in calculation.

**11.2.3** *Calculation* — as under **11.1.3.**

## 12. DETERMINATION OF ASH

### 12.1 Procedure

**12.1.1** Weigh 3 to 5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.01 g, char in a tared porcelain, silica or platinum crucible, and ignite at a low heat, not exceeding dull redness, until free from carbon and until the difference between successive weighings does not exceed 1 mg. Use a muffle furnace, if available.

**12.1.1.1** If a carbon-free ash cannot be obtained in this manner, extract the charred mass with hot water, collect the insoluble residue on an ashless

*Specification for rectified spirit ( *revised* ).

†An example of a suitable filter aid is diatomaceous material sold under the name of 'Filter-cel'.

filter paper, wash, and ignite the filter paper until all the carbon is consumed. Then transfer the filtrate and washings to the crucible, evaporate to dryness and heat to dull redness. Cool in a desiccator and weigh. Repeat until the difference between successive weighings does not exceed 1 mg. Use the lowest mass in the calculation.

### 12.2 Calculation

$$\text{Ash, percent by mass} = \frac{100\,m}{M}$$

where

$m$ = mass in g of ash, and

$M$ = mass in g of sample taken.

NOTE — For calculating results on moisture-free basis, *see* Note under **6.1.5.**

## 13. DETERMINATION OF MATTER SOLUBLE IN WATER

**13.0 Outline of the Method** — A known mass of powdered sample is digested with water, made up to a known volume and filtered. The mass that goes into solution is determined by evaporating an aliquot portion of the filtrate to constant mass and calculating for the whole solution.

### 13.1 Procedure

**13.1.1** Finely grind a sufficient quantity of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to pass 250-micron IS Sieve. Weigh 20 to 25 g of the powdered sample to an accuracy of 0.1 g and transfer to a beaker. Add 200 ml of distilled water and stir thoroughly. Cover the beaker with a watch glass and allow it to stand at 27 ± 1°C for 4 hours with occasional stirring.

**13.1.2** Filter into a 250-ml volumetric flask. Wash the residual sample and the filter paper with distilled water and make up to the graduation mark. Transfer a measured volume of the filtrate into a weighed evaporating dish and evaporate to dryness over a boiling water bath. Dry the residue to constant mass in an oven maintained at 100 ± 2°C.

**13.1.3** In case the acidity and alkalinity of the aqueous solution is to be tested, test small aliquots of the solution prepared in **13.1.2** with methyl red and bromothymol blue, respectively.

### 13.2 Calculation and Reporting

**13.2.1** Matter soluble in water, percent by mass $= \frac{M}{VM} \times 2{\cdot}5 \times 10^4$

where

$m$ = mass in g of residue,

$v$ = volume in ml taken for evaporation, and

$M$ = mass in g of sample taken.

NOTE — For calculating results on the basis of moisture-free sample, *see* Note under **6.1.5.**

**13.2.2** Report the acidity or alkalinity of the aqueous extract as observed in **13.1.3.**

## 14. DETECTION OF ORPIMENT

**14.0 Outline of the Method** — The presence of 0.5 percent or more of orpiment in a sample of shellac gives the shellac flake a yellow, opaque appearance. Even a trace of orpiment can be detected from a solution in spirit or in aqueous borax if the shellac is free from dirt.

### 14.1 Reagents

**14.1.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959* ); or 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959† ).

**14.1.2** *Borax Solution* — 5 percent ( by mass ) of borax ( $Na_2B_4O_7$, $10H_2O$ ) in distilled water.

### 14.2 Procedure

**14.2.1** Prepare in a small conical flask a solution of the sample ( strength 20 percent *m/v* ) in alcohol or borax solution. Allow the solution to stand when the orpiment settles in a layer at the bottom. Examine from below. Yellow particles of orpiment will be visible if it is present; 0.3 percent orpiment gives a continuous layer.

**14.2.1.1** This method is most sensitive when the alcohol is cooled to 0°C before preparing the solution. The same effect is not obtained by dissolving at room temperature and then cooling because, under these conditions, the agglomerates of wax are liable to hold the orpiment particles in suspension.

## 15. DETERMINATION OF ACID VALUE

**15.0 Outline of the Method** — An alcoholic solution of the sample is titrated with a solution of potassium hydroxide.

*Specification for rectified spirit ( *revised* ).

†Specification for ordinary denatured spirit ( *revised* ).

## 15.1 Method for Shellac

**15.1.1** *Reagents*

**15.1.1.1** *Alcohol* — 95 percent ( by volume ) rectified spirit ( conforming to IS : 323-1959* ); neutral.

**15.1.1.2** *Standard alcoholic potassium hydroxide solution* — 0.1 N. Check the strength of the alcoholic potash at intervals to provide for any deterioration in the strength of the alkali.

**15.1.1.3** *Thymol blue indicator* — Dissolve 0.10 g of thymol blue in 100 ml of alcohol.

**15.1.2** *Procedure* — Accurately weigh about 2 g of 'prepared sample' ( *see* **3.3.2** ) and dissolve in 50 ml of alcohol with slight warming, if necessary. Cool and carry out the titration with standard alcoholic potassium hydroxide solution using thymol blue as external indicator.

**15.1.3** *Calculation* — Express the acid value as the number of milligrams of potassium hydroxide required for 1 g of shellac

$$\text{Acid value} = 56{\cdot}1 \frac{VN}{M}$$

where

$V$ = volume in ml of standard potassium hydroxide solution required,

$N$ = normality of standard potassium hydroxide solution, and

$M$ = mass in g of sample taken.

## 15.2 Method for Bleached Lac

**15.2.1** *Reagents*

**15.2.1.1** *Phenolphthalein indicator* — Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ( by volume ) ethyl alcohol, and dilute with water to a final volume of 100 ml.

**15.2.1.2** *Neutral ethyl alcohol* — 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959† ) neutralized to phenolphthalein indicator.

**15.2.1.3** *Standard alcoholic potassium hydroxide solution* — 0.1 N in 95 percent ( by volume ) ethyl alcohol; alternatively,

*Standard aqueous potassium hydroxide or sodium hydroxide solution* — ( 0.1 N ), prepared as follows:

a) Prepare a stock concentrated solution by dissolving potassium hydroxide or sodium hydroxide in water in the proportion of 112 g

*Specification for rectified spirit ( *revised* ).

†Specification for ordinary denatured spirit ( *revised* ).

of potassium hydroxide or 85 g of sodium hydroxide in 200 ml of water. Allow the solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the carbonate precipitate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off 175 ml and dilute to 10 litres with water. Preserve in a bottle provided with a guard tube filled with soda lime.

b) Determine the normality by titrating against pure potassium acid phthalate, using phenolphthalein indicator. This solution will be approximately 0.10 N, but do not attempt to adjust it to any exact value. Determine its normality to ±0.001 N and make proper adjustment in using it.

**15.2.2** *Procedure* — Weigh 0.8 to 1.2 g of the 'prepared sample' ( *see* **3.3.3** ) to an accuracy of 1 mg and dissolve in 50 ml of neutral ethyl alcohol, dissolution being hastened by warming in hot water at 80 to 90°C for a few minutes. When solution is complete, cool and add 10 drops of phenolphthalein indicator. Titrate with standard alcoholic potassium hydroxide solution or with standard aqueous potassium hydroxide or sodium hydroxide solution.

**15.2.3** *Calculation* — Report the result in terms of milligrams of potassium hydroxide per gram of moisture-free sample calculated according to the formula:

$$\text{Acid value} = \frac{56{\cdot}10\ VN}{W}$$

where

$V$ = volume in ml of standard alkali solution used in the titration,

$N$ = normality of standard alkali solution, and

$W$ = mass in g of sample taken.

NOTE — For calculating results on the basis of moisture-free material, *see* Note under **6.1.5.**

## 16. DETERMINATION OF LEAD CONTENT

**16.0 Outline of the Method** — The lead content is determined colorimetrically by matching the colour of lead sulphide obtained from the material with that obtained from standard lead solution.

**16.1 Reagents** — The following reagents of analytical reagent grade are required. As far as possible, the reagents with the exception of **16.1.7.1** and **16.1.7.2** should be free from traces of lead.

**16.1.1** *Concentrated Sulphuric Acid* — sp gr 1.84 ( conforming to IS : 266-1961* ).

**16.1.2** *Citric Acid* — solid.

**16.1.3** *Ammonia* — sp gr 0.90 or diluted as required.

**16.1.4** *Potassium Cyanide Solution* — 10 percent ( *m/v* ).

**16.1.5** *Diphenylthiocarbazone* ( *Dithizone* ) *Solution* — 0.1 percent *( m/v* ) solution in chloroform, freshly prepared.

**16.1.6** *Dilute Hydrochloric Acid* — approximately 0.1 N.

**16.1.7** *Standard Lead Solution* — Two reference solutions of lead nitrate are required in this test as given below.

**16.1.7.1** *Standard strong lead solution* — obtained by dissolving 0.16 g of lead nitrate [ $Pb(NO_2)_2$ ] in 50 ml of dilute nitric acid and making up to 100 ml with water.

**16.1.7.2** *Standard dilute lead solution* — prepared freshly before the test, by diluting 1 ml of standard strong lead solution to 100 ml with water.

**16.1.8** *Ammonium Acetate* — solid.

**16.1.9** *Sodium Sulphide Solution* — 10 percent.

### 16.2 Procedure

**16.2.1** Take 4.5 to 5.5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) and char in a porcelain or silica basin at low heat not exceeding 500°C until free from carbon, taking care to avoid loss of the light ash. Cool, add 5 ml of water and 10 ml of hydrochloric acid and boil gently for 5 minutes. Cool and transfer the clear solution to a 100-ml, one-mark graduated flask, filtering through paper, if necessary. Make up the volume to 100 ml with water. Take an aliquot of the solution corresponding to 0.5 g of the sample originally taken. Add 5 ml of concentrated sulphuric acid and evaporate to fuming. Cool, dilute with about 50 ml of water, add 2 g of citric acid and just neutralize with ammonia. Add 1 ml of potassium cyanide solution and transfer the whole to a separating funnel. The total volume should be 100 to 150 ml.

**16.2.2** Extract the liquid with dithizone solution. Carry out three extractions using 10, 5 and 5 ml respectively, but if the last extraction gives any indication of a reddish tinge, extract again to ensure complete removal of lead.

**16.2.3** Take 10 ml of water in another separating funnel and wash each extract with this water. If suspended matter is present in the chloroform extract, this shall be filtered before passing to the separating funnel containing

*Specification for sulphuric acid ( *revised* ).

the 10 ml of wash water. Transfer the combined chloroform extracts to a separating funnel and extract lead by shaking successively with 50 ml, 20 ml and 10 ml of dilute hydrochloric acid. Combine the acid extracts in a separating funnel, wash once or twice with 10 ml of chloroform and filter through a previously wetted filter paper into a 100-ml graduated flask. Make up the volume of the filtrate to 100 ml with dilute hydrochloric acid and use this as the test solution.

**16.2.4** Estimate the lead present colorimetrically against the standard dilute lead solution containing 0.000 01 g of lead per ml ( using not more than 10 ml of standard solution for matching ) in the following manner.

**16.2.4.1** Transfer a suitable volume of the test solution to a Nessler cylinder. Add 2 g of ammonium acetate, followed by ammonia until just alkaline, and then 1 ml of potassium cyanide solution. Dilute to 50 ml, add 2 drops of sodium sulphide solution and match the colour against a set of standards prepared in the same way, using different volumes of standard lead solution.

**16.2.5** A blank determination shall be run under the same conditions, on the same reagents and by the same person but without using the material.

**16.3 Expression of Results** — Express the lead content as parts of lead per million parts of the sample.

## 17. DETERMINATION OF MINERAL ACID

### 17.1 Reagents

**17.1.1** *Neutral Ethyl Alcohol* — 95 percent ( by volume ) denatured spirit ( conforming to IS : 324-1959* ) neutralized to phenolphthalein indicator.

**17.1.2** *Sodium Chloride Solution* — 10 percent ( *m/v* ) solution.

**17.1.3** *Bromophenol Blue Indicator* — Warm 0.1 g of bromophenol blue with 30 ml of 0.05 N sodium hydroxide solution and 5 ml of 90 percent ( by volume ) ethyl alcohol; dilute the solution with 20 percent ( by volume ) ethyl alcohol to make the final volume 250 ml.

**17.1.4** *Standard Sodium Hydroxide Solution* — 0.1 N aqueous solution.

**17.1.5** *Potassium Hydrogen Phthalate* — containing not less than 99.9 percent and not more than the equivalent of 100.1 percent of potassium hydrogen phthalate ( $C_8H_5O_4K$ ) and impurities not exceeding 0.001 percent of chloride ( as Cl ), 0.01 percent of sulphate ( as $SO_4$ ), 0.1 percent of moisture, and 0.05 percent sodium ( Na ).

**17.1.6** *Buffer Solution* — *p*H 4.0. Dissolve 10.21 g of potassium hydrogen phthalate in freshly boiled and cooled water and make up the volume to 1 000 ml.

*Specification for ordinary denatured spirit ( *revised* ).

**17.2 Procedure** — Weigh 4.5 to 5.5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.01 g and dissolve by warming in 50 ml of neutral ethyl alcohol in a 500-ml conical flask. Cool the solution and add, with shaking, 200 ml of freshly boiled and cooled water, followed by 50 ml of sodium chloride solution. Shake intermittently over a period of one hour so that the lac is well coagulated. Allow to settle. Filter through hardened, close-texture filter paper ( Whatman No. 5 or equivalent ) using a Buchner funnel and filter flask, with the aid of a filter-pump; wash with two successive quantities of 25 ml of water. Transfer the filtrate and washings to a 400-ml tall form beaker, add 25 drops of bromophenol blue indicator and titrate with standard sodium hydroxide solution until the colour matches that of a standard consisting of 350 ml of buffer solution to which 15 drops of the same indicator have been added.

**17.3 Calculation** — Report the result to the nearest whole number, as the number of millilitres of 0.1 N sodium hydroxide solution required by 100 g of the moisture-free sample, according to the formula:

$$\text{Mineral acid} = \frac{VN \times 10^3}{M}$$

where

$V$ = number of ml of standard sodium hydroxide solution used in the titration,

$N$ = normality of standard sodium hydroxide solution, and

$M$ = mass in g of sample taken.

NOTE — For calculating results on the basis of moisture-free material, *see* Note under **6.1.5.**

## 18. TEST FOR TOTAL CHLORINE IN BLEACHED LAC

**18.0 Outline of the Method** — The bleached lac is treated with metallic sodium to convert the total chlorine into soluble chloride which is estimated volumetrically.

### 18.1 Reagents

**18.1.1** *Ethyl Alcohol* — 98 to 100 percent ( by volume ).

**18.1.2** *Metallic Sodium*

**18.1.3** *Dilute Nitric Acid* — 20 percent.

**18.1.4** *Silver Nitrate Solution* — approximately 0.1 N.

**18.1.5** *Nitrobenzene* — redistilled, free from chlorides.

**18.1.6** *Standard Potassium Thiocyanate Solution* — approximately 0.1 N, but of exactly the same normality as the silver nitrate solution ( *see* **18.1.4 ).**

**18.1.7** *Ferric Ammonium Sulphate Indicator* — a saturated solution acidified with nitric acid.

### 18.2 Procedure

**18.2.1** Weigh 0.4 to 0.6 g of the 'prepared sample' ( *see* **3.3.3** ) to an accuracy of 1 mg and dissolve in 50 ml of ethyl alcohol in a 250-ml conical flask. Reflux the solution using a 60 mm long condenser, while heating the flask on a hot plate. When all the lac is dissolved and the solution is boiling briskly, add slowly, piece by piece, through the top of the condenser, about 3 g of sodium freshly cut into small 3-mm cubes.

**18.2.2** When the reaction is complete and all the sodium is dissolved, allow the solution to cool. Remove the condenser, add 30 ml of water, and transfer the solution to a 500-ml conical flask, rinsing two or three times with a further 50 ml of water. Add a few glass beads to prevent bumping and evaporate the solution on a hot plate to about half the volume to remove ethyl alcohol. Cool and make slightly acidic with dilute nitric acid. Add from a burette 10 ml of silver nitrate solution and then add 2 ml of nitrobenzene and 1 ml of ferric ammonium sulphate solution. Stopper the flask and shake vigorously to coagulate the precipitate. Titrate the residual silver nitrate with standard potassium thiocyanate solution until a permanent faint reddish brown colouration appears. Carry out a blank determination using all reagents.

### 18.3 Calculation

$$\text{Total chlorine, percent by mass} = \frac{3{\cdot}546\,[\,V_1 - (V_2 - V_3)\,]\,N}{M}$$

where

$V_1$ = volume in ml of silver nitrate solution,

$V_2$ = volume in ml of standard potassium thiocyanate solution required in the blank test,

$V_3$ = volume in ml of standard potassium thiocyanate solution required for the sample,

$N$ = normality of silver nitrate solution, and

$M$ = mass in g of sample taken.

NOTE — For calculating results on the basis of moisture-free material, *see* Note under **6.1.5.**

## 19. TEST FOR FREE CHLORINE IN BLEACHED LAC

### 19.1 Reagent

**19.1.1** *Starch-Iodide Paper*

**19.2 Procedure** — Grind 5 g of the 'prepared sample' ( *see* **3.3.3** ) in a mortar with 20 ml of distilled water. Filter and test the filtrate for free chlorine with starch-iodide paper.

**19.2.1** If blue colour does not develop immediately, it shall be taken as indicating absence of free chlorine.

## 20. DETERMINATION OF ARSENIC

**20.0** Two methods for determination of arsenic content, one for appreciable amount of arsenic and the other for traces of arsenic, are described below. While both methods are applicable to shellac, only Method II is used for bleached lac.

### 20.1 Method I ( Appreciable Amount of Arsenic )

**20.1.0** *Outline of the Method* — The sample is digested with nitric acid and sulphuric acid, and the arsenic compound obtained by distillation after treating the solution with the chloride-hydrazine-bromide mixture is titrated by a suitable method.

**20.1.1** *Apparatus* — as shown in Fig. 5.

**20.1.2** *Reagents*

**20.1.2.1** *Concentrated nitric acid* — sp gr 1.42 ( conforming to IS : 264-1968* ).

**20.1.2.2** *Dilute nitric acid* — 30 parts by volume of concentrated nitric acid in 100 parts of solution.

**20.1.2.3** *Concentrated sulphuric acid* — sp gr 1.84 ( conforming to IS : 266-1961† ).

**20.1.2.4** *Chloride-hydrazine-bromide mixture* — Mix 5 g of sodium chloride, 0.5 g of hydrazine sulphate and 0.02 g of potassium bromide and store in a tightly stoppered bottle.

**20.1.2.5** *Concentrated hydrochloric acid* — sp gr 1.16 ( conforming to IS : 265-1962‡ ).

**20.1.2.6** *Methyl orange indicator* — 0.04 percent ( *m/v* ) solution in 20 percent ( *v/v* ) ethyl alcohol.

**20.1.2.7** *Standard potassium bromate solution* — 0.01 N.

**20.1.2.8** *Caustic soda solution* — approximately 2 N.

**20.1.2.9** *Sodium bicarbonate* — analytical grade.

**20.1.2.10** *Standard iodine solution* — 0.01 N.

*Specification for nitric acid ( *first revision* ).

†Specification for sulphuric acid ( *revised* ).

‡Specification for hydrochloric acid ( *revised* ).

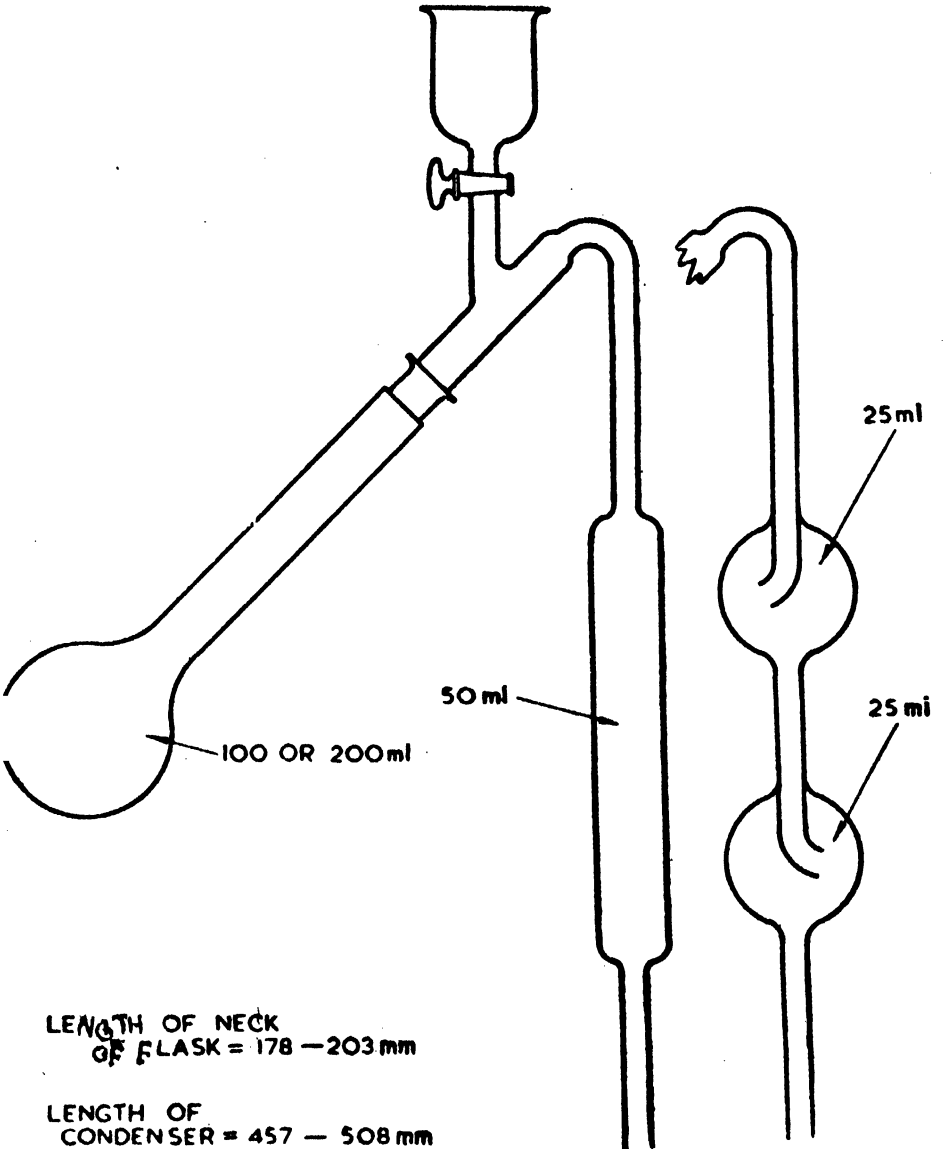



FIG. 5 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF ARSENIC, METHOD I

**20.1.2.11** *Starch solution* — Make a paste of 0.2 g of soluble ( potato ) starch in cold water and pour into 100 ml of boiling water. Boil for 5 minutes, cool and bottle. The solution should be prepared freshly every 2 or 3 days.

**20.1.3** *Procedure*

**20.1.3.1** Weigh 4.5 to 5.5 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.01 g and place with 10 ml of dilute nitric acid in a 100-ml or 200-ml resistant glass or silica Kjeldahl flask, and heat the mixture until any initial vigorous reaction subsides and ceases. Cool and add gradually 10 ml of concentrated sulphuric acid at such a rate as to prevent excessive frothing or heating (10 minutes are usually required) and continue heating. Add to the hot solution 5 ml of concentrated nitric acid in small portions, and boil until colourless. If necessary, add concentrated nitric acid in further small portions at a time. Note for the purpose of the blank determination the total amount of concentrated nitric acid added. ( The digestion usually takes about 30 minutes ). Cool, dilute with 50 ml of water and transfer to the flask of the distillation apparatus. Boil the solution, without inserting the condensing arm, till the bulk is reduced to about 10 ml or until white fumes appear; cool, dilute and again boil down to 10 ml; cool and add 7 ml of water. Cool well the liquid, add 5 g of the chloride-hydrazine-bromide mixture, followed rapidly by 10 ml of concentrated hydrochloric acid.

**20.1.3.2** Fit the condenser quickly and distil the liquid into 20 ml of water, the exit tube dipping below the surface of the liquid; cool in ice until about 5 minutes after the condenser is full of steam. Dilute the distillate to 100 ml, add methyl orange indicator, heat the solution to 80°C, and titrate with standard potassium bromate solution, or, alternatively, nearly neutralize the distillate with caustic soda solution, then add 3 g excess sodium bicarbonate and titrate the solution with standard iodine solution using starch as indicator.

**20.1.3.3** Make sure that no solid material comes in contact with the ground-in portion of the flask.

**20.1.3.4** A blank determination shall be run under the same conditions, on the same reagents and by the same person but without using the material.

**20.1.4** *Calculation* — Express the arsenic content of the sample as percentage by mass of arsenious sulphide ( $As_2S_3$ ).

$$\text{Arsenious sulphide } (As_2S_3), \text{ percent by mass} = \frac{6{\cdot}15\,VN}{M}$$

where

$V$ = volume in ml of standard iodine solution, or standard potassium bromate solution, required in the titration;

$N$ = normality of standard iodine solution, or standard potassium bromate solution, employed in the titration, and
$M$ = mass in g of sample taken.

## 20.2 Method II ( Traces of Arsenic )

**20.2.1** *Apparatus* — assembled as shown in Fig. 6.

**20.2.1.1** *Wide-mouth bottle* — capacity 120 ml.

**20.2.1.2** *Glass tube* — made from ordinary glass tubing, and having a total length of 200 mm. It should have an internal diameter of exactly 6.5 mm and an external diameter of about 8 mm. It is drawn out at one end to a diameter of about 1 mm and a hole not less than 2 mm in diameter is blown in the side of the tube, near the constricted part. The upper end of the tube is cut off square and is either rounded off slightly or ground smooth.

**20.2.1.3** *Rubber bungs* — three. One fits exactly into the mouth of the wide-mouth bottle and has a hole bored centrally to take the tube from its constricted end. Each of the other two rubber bungs ( about 25 × 25 mm ) has a hole, exactly 6.5 mm in diameter, bored centrally and are fitted with a rubber band or spring clip for holding them tightly together.

**20.2.1.4** *Preparation of the glass tube* — Moisten a small quantity of cotton wool with lead acetate solution and then dry it in a dust-free atmosphere. Lightly pack the glass tube with this cotton wool, so that the upper surface of the cotton wool is not less than 25 mm below the top of the tube. Insert the upper end of the tube into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm when the tube has a rounded off end, or so that the ground end of the tube is flush with the larger end of the bung. Place a piece of mercuric chloride paper flat on the top of the bung. Place the other bung over this with its larger end in contact with the piece of mercuric chloride paper. Fasten the two bungs by means of the rubber band or the spring clip in such a manner that the borings of the two bungs ( or the upper bung and the glass tube ) meet to form a true tube of 6.5 mm diameter interrupted by a diaphragm of mercuric chloride paper. Instead of this method of attaching the mercuric chloride paper, any other method may be used, provided (a) that the whole of the evolved gas passes through the paper; (b) that the portion of the paper in contact with the gas is a circle of 6.5 mm diameter; and (c) that the paper is protected from sunlight during the test.

**20.2.2** *Reagents* — The following reagents are required. The reagents with the exception of those in **20.2.2.13** and **20.2.2.14** should be free from traces of arsenic.

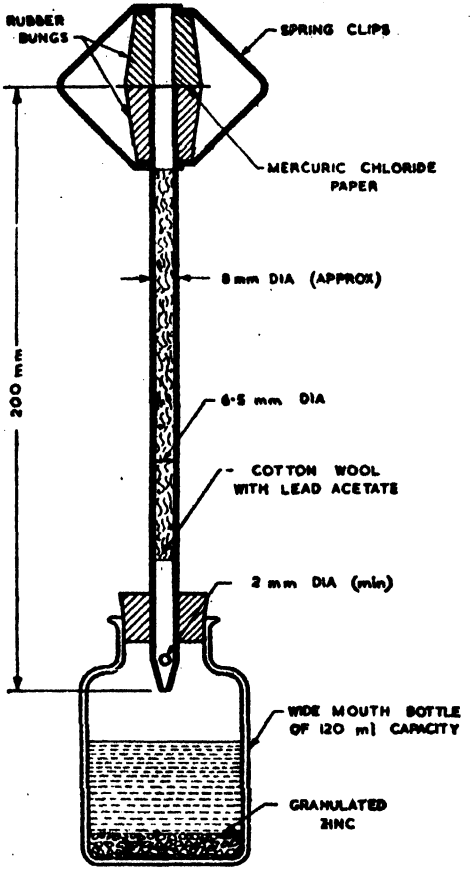



FIG. 6 APPARATUS FOR THE DETERMINATION OF ARSENIC, METHOD II

**20.2.2.1** *Concentrated nitric acid* — sp gr 1.42 ( conforming to IS : 264-1968* ).

**20.2.2.2** *Dilute nitric acid* — 30 parts by volume of concentrated nitric acid in 100 parts of the solution.

**20.2.2.3** *Concentrated sulphuric acid* — sp gr 1.84 ( conforming to IS : 266 - 1961† ).

*Specification for nitric acid *(first revision* ).

†Specification for sulphuric acid ( *revised )*.

**20.2.2.4** *Chloride-hydrazine-bromide mixture* — Mix 5 g of sodium chloride, 0.5 g of hydrazine sulphate and 0.02 g of potassium bromide, and store in a tightly stoppered bottle.

**20.2.2.5** *Concentrated hydrochloric acid* — sp gr 1.16 ( conforming to IS : 265-1962* ).

**20.2.2.6** *Methyl orange indicator* — 0.04 percent ( *m/v* ) solution in 20 percent (*v/v*) ethyl alcohol.

**20.2.2.7** *Lead acetate solution* – 100 percent (*m/v*) in distilled water, recently boiled.

**20.2.2.8** *Mercuric chloride paper* — smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of mercuric chloride in water, pressed to remove superfluous solution, and dried at 60°C in the dark. The grade of the filter paper shall be such that the mass in $g/m^2$ shall be between 65 and 120; the thickness in mm of 500 papers shall be approximately equal, numerically, to the mass in $g/m^2$. Mercuric chloride paper should be stored in a stoppered bottle in the dark. Papers which have been exposed to sunlight or to the vapour of ammonia should not be used as they give a lighter coloured stain or no stain at all when employed in the quantitative test for arsenic.

**20.2.2.9** *Stannous chloride solution* — Dilute 60 ml of concentrated hydrochloric acid with 20 ml of water, add to it 20 g of tin, heat gently until gas ceases to be evolved, and add sufficient water to produce 100 ml, allowing the undissolved tin to remain in the solution. Decant the clear solution, add an equal volume of concentrated hydrochloric acid, boil down to the original volume and filter through a fine-grained filter paper.

**20.2.2.10** *Stannated hydrochloric acid* — Mix together 1 ml of stannous chloride solution and 100 ml of concentrated hydrochloric acid.

**20.2.2.11** *Potassium iodide* — crystals or in the form of powder.

**20.2.2.12** *Zinc* — granulated, complying with the following test:

Take 50 ml of water, 10 ml of stannated hydrochloric acid and 0.1 ml of dilute solution of arsenic ( *see* **20.2.2.14** ) in the wide-mouth bottle. Add 1 g of potassium iodide and 10 g of zinc. Quickly place the prepared glass tube ( *see* **20.2.1.4** ) in position. Allow the reaction to continue for 1 hour. A faint but distinct yellow stain shall be produced on the mercuric chloride paper.

**20.2.2.13** *Strong solution of arsenic* — Dissolve 0.132 g of arsenic trioxide in 50 ml of concentrated hydrochloric acid and add sufficient water to produce 100 ml.

*Specification for hydrochloric acid ( *revised* ).

**20.2.2.14** *Dilute solution of arsenic* — freshly prepared. Dilute 1 ml of strong solution of arsenic with water sufficient to produce 100 ml. This solution contains 0.01 mg of arsenic ( or 0.013 2 mg of $As_2O_3$ ) per ml.

**20.2.3** *Procedure*

**20.2.3.1** Treat 5 g of the sample exactly in the manner prescribed in **20.1.3.1** and then proceed as follows.

**20.2.3.2** Fit the condenser quickly and distil the liquid into a mixture of 10 ml of water and 2 mi of concentrated nitric acid. Then evaporate the distillate to dryness on the water bath and evaporate the residue twice to dryness with 5 ml of water to remove nitric acid. Dissolve the final residue by warming in 3 ml of concentrated sulphuric acid, cool and dilute with water. Transfer the whole of the solution to the wide-mouth bottle, add 15 ml of stannated hydrochloric acid and 1 g of potassium iodide. Then add 10 g of zinc. Quickly place the prepared glass tube ( *see* **20.2.1.4** ) in position. Allow the reaction to continue for 40 minutes. Remove the piece of mercuric chloride paper at the end of this period. If arsenic is present in the material, compare the yellow stain produced on the mercuric chloride paper, by daylight, with the standard stains prepared as described under **20.2.3.4.** If the stain in this test exceeds that equivalent to 0.02 mg of arsenious oxide ( $As_2O_3$ ), make the solution to a known bulk with dilute sulphuric acid ( 1 : 8 ) and take an aliquot to produce a stain suitable for matching. The reaction may be accelerated by placing the apparatus on a warm surface, care being taken that the mercuric chloride paper remains quite dry throughout the test. The most suitable temperature for carrying out the test is generally about 40° C, but because the rate of evolution of the gas varies somewhat with different batches of zinc, the temperature may be adjusted to obtain a regular, but not too violent, evolution of gas. The tube should be washed with concentrated hydrochloric acid, rinsed with water, and dried between successive tests.

**20.2.3.3** *Comparison of stains* — The comparison of the stains is made with freshly prepared standard stains immediately at the completion of the test.

**20.2.3.4** *Preparation of standard stains* — Mix together 50 ml of water, 10 ml of stannated hydrochloric acid and appropriate volumes of dilute solution of arsenic. Treat the resulting solutions as described under **20.2.3.2** to prepare the standard stains.

**20.2.3.5** Make sure that no solid material comes in contact with the ground-in portion of the bottle.

**20.2.3.6** A blank determination shall be carried out under the same conditions, on the same reagents and by the same person but without using the material. The blank should not produce any visible stain on the mercuric chloride paper.

**20.2.4** *Calculation* — Express the arsenic content of the sample as parts of arsenic ( As ) or arsenious oxide ( $As_2O_3$ ) per million parts of the sample.

## 21. FLOW TEST FOR SHELLAC

**21.0 Outline of the Method** — The method consists in melting a sample of ground shellac in a graduated test- tube or a plain test-tube, and then tilting the tube to an angle of 15 degrees, while maintained at 100 ± 1°C, in order to permit the shellac to flow down the tube, and measuring (a) the time required for the shellac to flow to the various graduations along the test-tube, or (b) the total distance the shellac flows along the test-tube in a specified time.

### 21.1 Test Specimens

**21.1.1** Test two specimens for each of the methods.

**21.1.2** For each test specimen, accurately weigh 2.00 g of 'prepared sample' ( *see* **3.3.2** ). Spread it out in a shallow vessel and place in a desiccator over a saturated solution of sodium dichromate with an excess of solid salt and leave in this atmosphere at room temperature for at least 24 hours. Test the specimens immediately upon removal from the desiccator.

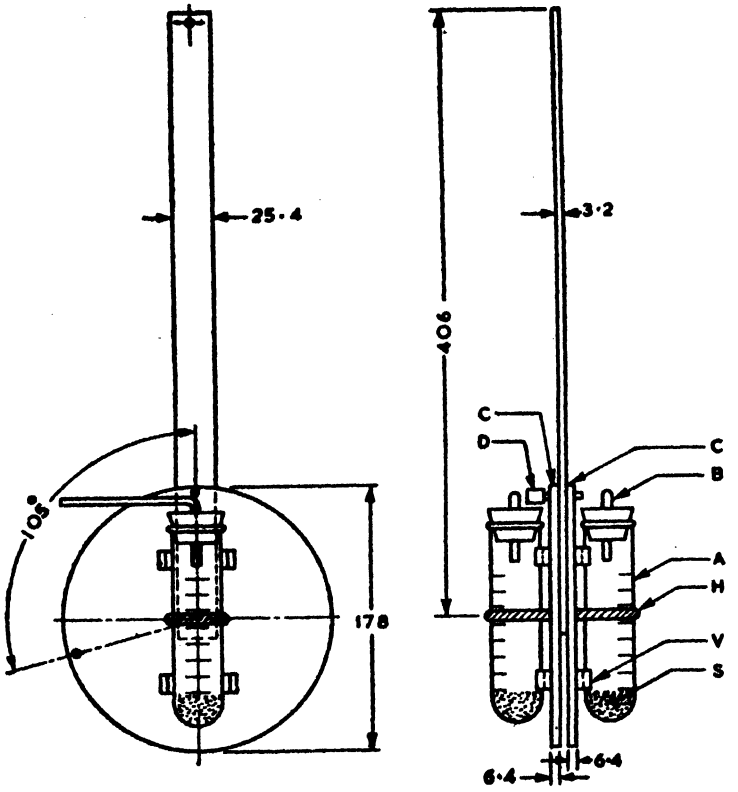
### 21.2 Method A

**21.2.1** *Apparatus* — The apparatus ( *see* Fig. 7 and 8 ) consists of the following.

**21.2.1.1** *Test-tubes* — two test-tubes *A* for holding the sample, preferably made of heat-resistant glass, red lined, 130 mm in length, 25 mm in outside diameter and 1.5 mm in wall thickness, graduated in 5 mm divisions beginning 11 mm from the outside bottom and extending upwards to 100 mm, every 10 mm line being numbered. Stopper the test-tubes with tightly fitting corks, with small breather tubes *B*.

**21.2.1.2** *Support* — a fixture *C* for holding the glass tubes in the correct position. It consists of two brass discs supported as a pendulum as indicated in Fig. 7 and 8, the discs being free to turn on the supporting shaft. These discs are held in the desired position by the pin *D,* and the tubes are supported on narrow V-blocks and held in position by coil spring *H* attached to discs *C*.

**21.2.1.3** *Oil bath* — a glass tank *E* heated by an electric immersion heater. Glycerine or a clear oil having a kinematic viscosity of 31 cst ( approximately ) is considered satisfactory for the bath. It is essential to use a mechanical stirring device *F* to maintain a uniformly distributed temperature.




All dimensions in millimetres.

FIG. 7 FLOW TEST APPARATUS FIXTURE SHOWING ESSENTIAL PARTS WITH TEST-TUBES IN VERTICAL POSITION

**21.2.1.4** *Thermometers — G* shall be partial immersion type graduated in degrees centigrade. A thermometer having a range of — 7°C to 300°C, with subdivisions at every 1°C, longer graduation at every 5°C and numbered graduations at eacn multiple of 10°C, is considered satisfactory for the purpose.

**21.2.2** *Procedure*

**21.2.2.1** Place the two specimens of shellac, each weighing 2.00 g in separate glass test-tubes, care being taken that the top surface of the specimen in each tube is level and at right angles to the walls of the tube and

that none of the powdered shellac adheres to the walls. Read the top level of the dry shellac in each tube on the millimetre graduated scale. Clamp the tubes containing the specimens in place in the testing fixture ( *see* Fig. 7 ).

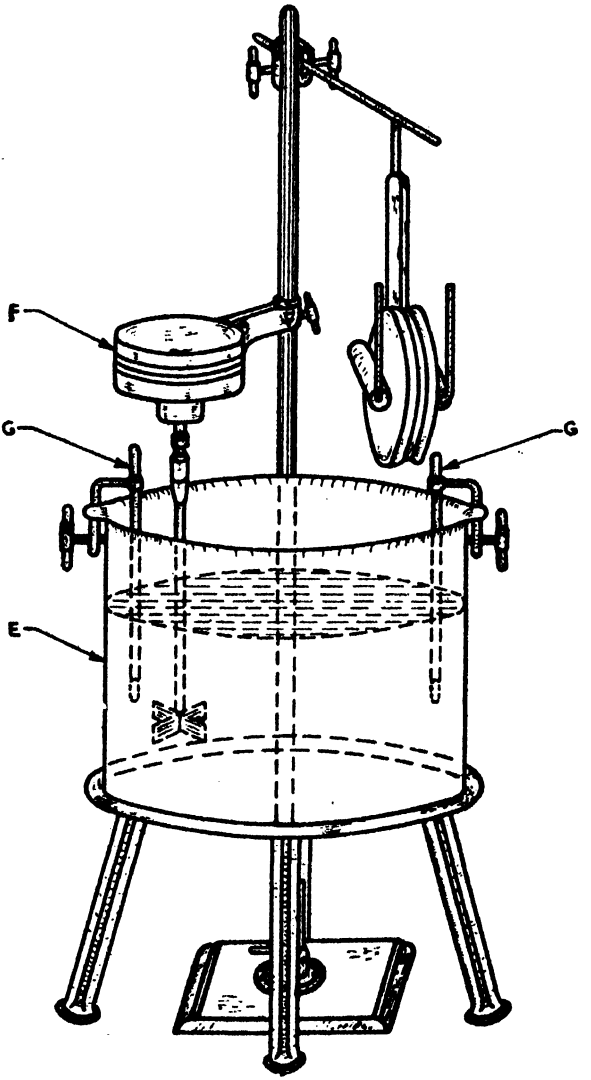



FIG. 8 FLOW TEST APPARATUS, ARRANGED WITH TUBES IN INCLINED POSITION FOR IMMERSION IN BATH

Insert the testing fixture, with the glass test-tubes in a vertical position, in the oil bath maintained at the test temperature of 100 ± 1°C. Allow the specimen to melt for 3 minutes.

**21.2.2.2** At the end of the 3-minute melting period, place each test-tube at an angle of 15 degrees from the horizontal, with the corked ends down ( *see* Fig. 8 ), and with the breather tubes extending above the level of the oil bath. Make the change from the vertical to the flow position as quickly as possible. With the oil bath maintained at 100 ± 1°C, record the total time required for the shellac in each tube to flow from the initial level of the shellac to each 10 mm marking along the tube. Discontinue the test in each tube when the flow is 90 mm or the total time is 20 minutes.

**21.2.3** *Report* — The report of the test shall include the following:

a) The time required for each 10-mm distance of flow for each specimen.

b) A graph showing the data reported in (a) above, with time plotted as abscissa and distance of flow as ordinate.

c) The angle of the test-tubes during the flowing period.

d) The atmospheric temperature and humidity of the laboratory.

## 21.3 Method B

**21.3.1** *Apparatus* — Any apparatus that will provide for accurately maintaining the required test temperature and the required positions of the test-tube may be used, but a suitable apparatus of the type shown in Fig. 7 and 8 consists of the following.

**21.3.1.1** *Test-tubes* — same as described under **21.2.1.1.**

**21.3.1.2** *Support* — same as described under **21.2.1.2.**

**21.3.1.3** *Oil bath* — same as described under **21.2.1.3** except that a metal or any other suitable container may be used and the heating may be by a Bunsen burner or an electric immersion heater.

**21.3.1.4** *Thermometers* — same as described under **21.2.1.4.**

**21.3.2** *Procedure*

**21.3.2.1** Prepare the apparatus and proceed with the test as described under **21.2.2.1.**

**21.3.2.2** At the end of the 3-minute melting period, place the test-tubes at an angle of 15 degrees from the horizontal, with the corked ends down ( *see* Fig. 8 ) and with the breather tube extending above the level of the oil bath. Make the change from the vertical position to the flow position as quickly as possible. With the oil bath maintained at the test temperature of 100 ± 1°C, allow the test-tubes to remain in the bath in this position for exactly 12 minutes. Remove the test-tubes immediately from the bath,

place in a vertical position, cool, wipe, and measure the flow of shellac in each tube by reading the distance between the initial point and the end of the flow tongue. Disregard the 'feather' caused by separation of wax from the shellac, at the very tip of the tongue.

NOTE — The 'feather' can be distinguished from the main body of shellac as it is always of a different colour.

**21.3.3** *Report* — The report of test shall include the following:

a) The flow expressed in millimetres for each specimen,
b) The average of the values in (a) above,
c) The angle of the test-tubes during the flowing period, and
d) The atmospheric temperature and humidity of the laboratory.

## 22. HEAT POLYMERIZATION TEST FOR SHELLAC

**22.0 Outline of the Method** — The method consists in heating shellac under specified conditions in a test-tube and observing the time required for it to attain a rubbery state as indicated by the 'spring-back' of a glass rod when it is twisted through a full circle.

**22.1 Apparatus** — The apparatus shown in Fig. 9 consists of the following parts.

**22.1.1** *Test-Tube* – glass test-tube 150 mm long and 25 mm inner diameter, placed in a suitable rack for holding test-tubes in a vertical position in an oil bath.

**22.1.2** *Glass Rod* — A smooth glass rod, about 210 mm long and 10 mm diameter, having a smaller glass rod, 20 mm long and 5 mm diameter attached to it at right angles about 180 mm from bottom end of the main rod. Bottom end of the main rod and the free end of the projection rod shall be smoothly rounded to a hemisphere of the respective rod diameters.

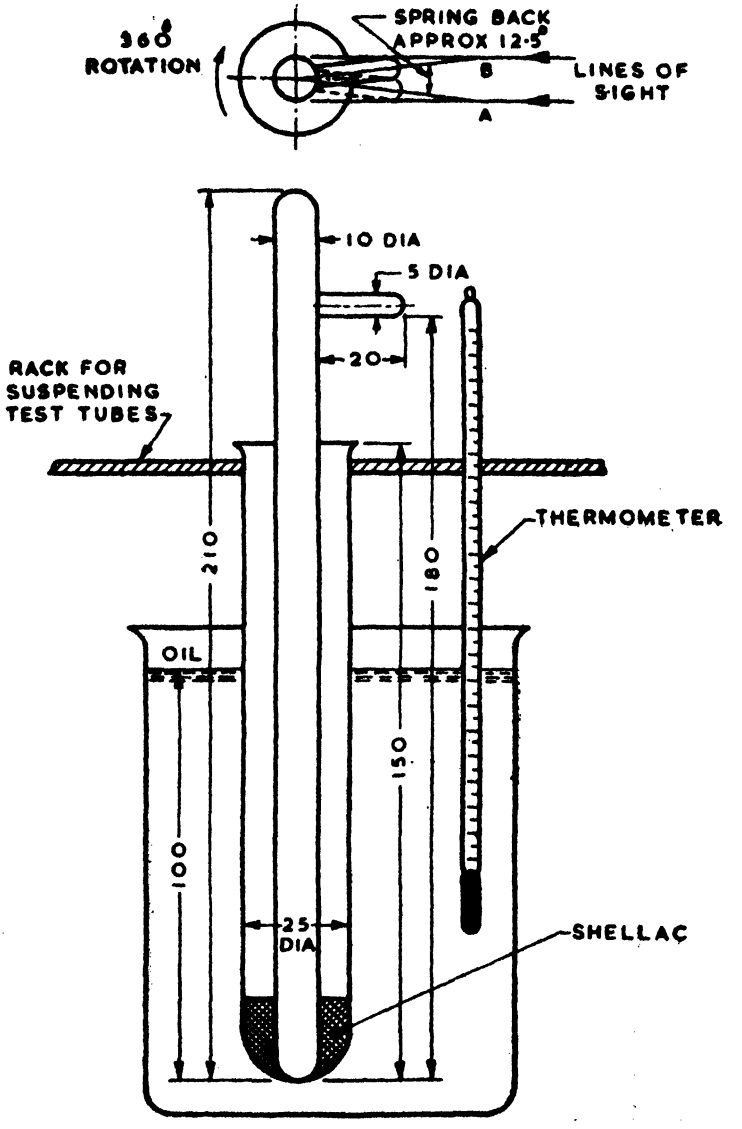
**22.1.3** *Oil Bath*—provided with means for continuous stirring and of such construction that it can be maintained within ± 1°C of the specified test temperature, which unless otherwise agreed, shall be 150°C.

**22.1.4** *Thermometer* — a suitable thermometer to indicate the test temperature.

**22.2 Conditioning** — Before testing, roll about 12 g of the 'prepared sample' ( *see* **3.3.2** ) on a clean filter paper and then carefully dry it. For doing this, place a flat bottom dish about 1 00 mm in diameter, containing the sample of shellac well spread out, in a well-ventilated oven at a temperature of 41 ± 1°C for approximately 16 hours. Immediately after taking it out from the oven, transfer the sample to a clean, dry bottle. Stopper the bottle tightly and allow the sample of shellac to cool in the bottle. Do not open the bottle except when removing a specimen for test.

**22.3 Specimens**

**22.3.1** Test two specimens of 5.0 g each for each sample of shellac.




NOTE — Mechanical stirrer not shown.
All dimensions in millimetres.

FIG. 9 HEAT POLYMERIZATION TEST APPARATUS

**22.4 Procedure** — Weigh 5.0 g of the 'conditioned' sample of shellac and transfer to the test-tube along with the glass rod. Insert the tube in the test rack so that it is held in a vertical position, dipped to a depth of about 100 mm in the oil bath maintained thermostatically at the specified temperature within ± 1°C. Record the time when the test-tube enters the oil bath. Holding the top of the test-tube with one hand, stir the shellac gently with the other hand for the first three minutes of the test, with a rotatory motion of the glass rod, so that the rod moves near the wall of the test-tube, and the shellac gets melted in as short a time as possible. At the end of each minute thereafter, turn the rod through a full circle ( about 360° ), and standing at arm's length away from the rod, take a line of sight so that when viewed with one eye, one edge of the main rod is in line with one edge of the top of the projection rod ( Fig. 9, Position A ); then release the rod forthwith and allow it to spring-back, keeping the line of sight fixed. If the rod springs back so far as to bring the other edge of the main rod in line with the opposite edge of the top of the projection rod ( Fig. 9, Position B ), assume that the end point has been reached. This much angular movement corresponds to about 12.5 degrees of spring-back which is arbitrarily fixed for the purpose of controlled observations as indicating the end point for noting the time for the heat polymerization test. Note the time when the end point is reached. Note the oil temperature every minute during the test to ensure that it does not vary beyond the specified limit of ± 1°C.

**22.5 Report** — The report of test shall include the following:

a) The test temperature,

b) The polymerization time to the nearest minute for each specimen, and

c) The mean of the readings under (b) above.

## 23. DETERMINATION OF GRIT IN SHELLAC

**23.0 Outline of the Method** — The residue obtained on 63-micron IS Sieve after alkaline dissolution of shellac and subsequent treatment with aqua regia, is dried and determined as grit.

### 23.1 Reagents

**23.1.1** *Aqua Regia* — Add 1 volume of concentrated nitric acid to 3 volumes of concentrated hydrochloric acid.

**23.1.2** *Sodium Carbonate, Anhydrous*

**23.1.3** *Sodium Carbonate Solution* — 1 percent ( *m/v* ) of the anhydrous material.

### 23.2 Procedure

**23.2.1** To 25 g of the 'prepared sample' ( *see* **3.3.2** ) contained in a 600-ml tall beaker, add 400 ml of hot water containing 7 g of anhydrous sodium

carbonate. Immerse the beaker in a hot water-bath at 92.5 ± 2.5°C and stir frequently over a period of 1 hour to ensure that the material has dissolved as far as possible. . Allow the beaker and the contents to cool and stand undisturbed for at least half-an-hour, then carefully remove the turbid supernatant liquor to within 2.5 cm of the bottom of the beaker, using a siphon or suction tube with a hooked end.

**23.2.2** Dilute the remaining liquor with 300 ml of the sodium carbonate solution, mix, allow to settle, and remove the supernatant liquor as before. Repeat this process twice more. Strain the remaining contents of the beaker through a 63-micron IS Sieve, transfer any matter remaining in the beaker to the sieve by means of a jet of water and wash well with water.

**23.2.3** Dry any residue on the sieve in an oven at 100 ± 2°C and transfer the residue to a porcelain crucible, ignite at a dull red heat and allow to cool. Heat the residue with 5 ml of aqua regia in a boiling water-bath for 30 minutes. Dilute the acid mixture with water and filter through an acid-resistant filter paper. Transfer any solid matter to the filter by means of a jet of hot water and wash until free from acid. Carefully fold and dry the washed filter paper and ignite in a platinum or procelain crucible at dull red heat. Sieve the residue through 63-micron IS Sieve and weigh any solid matter retained.

### 23.3 Calculation

$$\text{Grit, percent by mass} = \frac{100\ M_1}{M}$$

where

$M_1$ = mass in g of material retained on the sieve, and

$M$ = mass in g of the sample taken.

## 24. DETERMINATION OF IODINE VALUE

**24.0** Two alternative methods are described below.

### 24.1 Method I ( Hübl Method )

**24.1.1** *Reagents*

**24.1.1.1** *Rectified spirit* — conforming to IS : 323-1959*.

**24.1.1.2** *Hübliodine solution* — Mix together equal volumes of a 5 percent ( *m/v* ) solution of iodine in rectified spirit and a 6 percent ( *m/v* ) solution of mercuric chloride in rectified spirit. Allow to stand for at least 6 hours before use, but discard if more than 48 hours old.

*Specification for rectified spirit ( *revised* ).

**24.1.1.3** *Potassium iodide solution* — 10 percent ( *m/v* ) aqueous solution.

**24.1.1.4** *Sodium thiosulphate solution* — 0.1 N, standardized.

**24.1.1.5** *Starch solution* — 0.1 percent ( *m/v* ), freshly prepared.

**24.1.2** *Procedure*

**24.1.2.1** Weigh accurately about 0.6 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) into a 250-ml heat-resistant glass flask provided with a ground-glass stopper. Dissolve the sample in 10 ml of rectified spirit with the aid of gentle heat. Cool and add from a pipette 20 ml of the Hübl iodine solution. In a similar flask place 10 ml of rectified spirit and 20 ml of Hübl iodine solution to serve as a blank.

**24.1.2.2** Place the bottles in a cool, dark cupboard and leave for 16 to 18 hours ( preferably overnight ). Add 10 ml of potassium iodide solution to the contents of each bottle, rinsing the stopper and neck of the bottle to dissolve any iodine which may have tended to sublime. Dilute with 100 ml of water and titrate the excess iodine with sodium thiosulphate solution, using the starch indicator in the usual manner, vigorously shaking the contents of the bottle before and during the titration so as to break up the clots formed by the addition of water.

**24.1.3** *Calculation*

$$\text{Hübl iodine value} = \frac{1{\cdot}269\ (T_2 - T_1)}{M}$$

where

$T_2$ = volume in ml of 0.1 N sodium thiosulphate solution required for blank,

$T_1$ = volume in ml of 0.1 N sodium thiosulphate solution required for test, and

$M$ = mass in g of the sample taken.

## 24.2 Method II (Wij's-Langmuir Method)

**24.2.1** *Reagents*

**24.2.1.1** *Acetic acid* — glacial, 99 percent, having a melting point of 14.8 ± 0.05°C, and free from reducing impurities as indicated by the test given below:

Dilute 2 ml of acetic acid with 10 ml of water, add 0.1 ml of 0.1 N potassium permanganate solution and maintain at 27 ± 2°C. At the end of 2 hours, the pink colour shall not be discharged.

**24.2.1.2** *Chloroform*

**24.2.1.3** *Sodium thiosulphate solution* — 0.1 N, standardized.

**24.2.1.4** *Starch solution* — 0.1 percent ( *m/v* ) freshly prepared.

**24.2.1.5** *Potassium iodide solution* — 10 percent (*m/v*) aqueous solution.

**24.2.1.6** *Wij's-Langmuir iodine monochloride solution*

a) Dissolve 8 g of iodine trichloride in 500 ml of glacial acetic acid. Place 5 ml of the solution, accurately measured, in a glass-stoppered flask containing 10 ml of 10 percent aqueous potassium iodide solution. Add 100 ml of distilled water and titrate with 0.1 N sodium thiosulphate solution using starch as indicator. Calculate the exact quantity of iodine trichloride present in the solution from the factor:

1 ml of 0.1 N sodium thiosulphate = 0.005 832 g of $ICl_3$.

b) Dissolve 9 g of iodine in 500 ml of glacial acetic add, heating if necessary. Determine the exact quantity of iodine present by titrating 10 ml of this solution with 0.1 sodium thiosulphate solution using starch indicator.

c) One gram iodine trichloride reacts with 1.088 g of iodine to form iodine monochloride. From this relation calculate the volume of the iodine solution required to be added to the iodine trichloride solution. Add this calculated volume of iodine solution to iodine trichloride solution and mix thoroughly. Dilute this solution with glacial acetic acid till 10 ml is equivalent to 20 ml of 0.1 N sodium thiosulphate solution when it is titrated in the presence of excess of aqueous potassium iodide and water using starch as indicator.

d) Heat the solution thus prepared to 100°C for 10 minutes and then allow to cool. During the preparation of the solution prevent access of water vapour. Keep the solution in an amber coloured receptacle protected from light.

**24.2.2** *Procedure*

**24.2.2.1** Weigh about 0.2 g of the 'prepared sample' ( *see* **3.3.2** and **3.3.3** ) to an accuracy of 0.001 g and introduce into a 250-ml dry, clear glass bottle having a ground-glass stopper.

**24.2.2.2** Add 20 ml of acetic acid into the bottle and place it on the top of a hot water-bath at 67.5 ± 2.5°C, swirling the bottle occasionally until the solution is complete, except for the wax. This should not require more than 15 minutes. Add 10 ml of chloroform and cool the solution to 22 ± 0.5°C. Before adding the Wij's-Langmuir solution, allow the bottle to stand at a temperature of 22 ± 0.5°C for at least 30 minutes, half immersed in water in shallow pan of water which is either well-insulated or equipped with a suitable thermostat. Add 20 ml of Wij's-Langmuir solution at a temperature of 22 ± 0.5°C from a standard pipette with a delivery time of approximately 30 seconds. Immediately close the bottle, place it again in the

pan of water and note the time. Keep the bottle half immersed in water at 22 ± 0.5°C for 60 minutes, swirling the bottle occasionally during this time. Add 10 ml of the potassium iodide solution into the bottle and wash into it any Wij's-Langmuir solution left on the stopper. Dilute with about 100 ml of water and titrate the solution immediately. Run in rapidly 25 to 30 ml of standard sodium thiosulphate solution and shake vigorously until the solution assumes a straw colour. Add 1.5 ml of starch solution and slowly finish the titration. The end point is sharp. Disregard any colour returning after about 30 seconds.

**24.2.2.3** If several samples are being tested, allow at least 5 minutes interval between the additions of Wij's-Langmuir solution to different bottles to allow time for the final titration.

**24.2.2.4** Perform a blank determination under the same conditions and on the same reagents but omitting the sample.

### 24.3 Calculation

$$\text{Iodine value} = \frac{1{\cdot}269\,(T_3 - T_1)}{M}$$

where

$T_3$ = volume in ml of 0.1 N sodium thiosulphate solution required for blank,

$T_1$ = volume in ml of 0.1 N sodium thiosulphate solution required for test, and

$M$ = mass in g of the sample taken.

## 25. TEST FOR CLARITY OF SHELLAC SOLUTION

### 25 .1 Apparatus

**25.1.1** *Glass Measuring Cylinder* — stoppered, 200-ml capacity.

### 25.2 Reagent

**25.2.1** *Rectified Spirit* — conforming to IS : 323-1959*.

**25.3 Procedure** — Place 10 g of the 'prepared sample' ( *see* **3.3.2** ) in the glass-stoppered cylinder. Add 100 ml of rectified spirit and shake vigorously. Maintain at room temperature, shake at intervals until all soluble material has dissolved or up to a maximum of four hours, whichever is earlier. Allow the solution to stand undisturbed for 10 minutes and examine for absence of turbidity.

*Specification for rectified spirit ( *revised* ).

## 26. DETERMINATION OF BLEACH INDEX AND BLEACHABILITY OF SEEDLAC

**26.0** Two methods are described. Method I ( ILRI Method ) is the one recommended for adoption in India. It is based upon the investigations conducted by Y. Sankaranarayanan and P. K. Bose at the Indian Lac Research Institute and described in the Journal of Scientific & Industrial Research, Vol 13B, 1954; p 506. Method II is in vogue in USA.

### 26.1 Method I ( for Bleach Index and Bleachability )

**26.1.0** *Outline of the Method* — In the method described, the same amount of bleach liquor is added to all samples of lac irrespective of quality or grade and the degree of bleaching effected in each case is determined from the colour of the ( filtered ) bleached solution measured with the help of a photo-electric colorimeter which has been previously calibrated in terms of bleach index with the aid of iodine solution.

**26.1.1** *Apparatus*

**26.1.1.1** *Beakers* — tall, of 400-ml capacity.

**26.1.1.2** *Graduated measuring cylinder* — unstoppered, of 300- or 500-ml capacity.

**26.1.1.3** *Photo-electric colorimeter*

**26.1.2** *Reagents*

**26.1.2.1** *Sodium carbonate* — anhydrous, analytical reagent grade.

**26.1.2.2** *Phenolphthalein indicator* — Dissolve 0.1 g of phenolphthalein in 60 ml of ethyl alcohol ( 95 percent by volume ), and dilute with water to a final volume of 100 ml.

**26.1.2.3** *Bleach liquor* — A solution of sodium hypochlorite containing 3.00 ± 0.05 percent available chlorine, prepared from analytical quality reagent; alternatively it may be prepared from pure bleaching powder*, or sodium hydroxide as described below:

a) *From bleaching powder* — Weigh sufficient quantity of bleaching powder to give a little more than one litre of a 3 percent available chlorine solution. Triturate this powder with small portions of water, transferring them to a 2 000-ml beaker, until a volume of about 600 ml is obtained. Prepare a 15 percent solution of sodium carbonate and add this in small quantities to the bleaching powder solution, stirring thoroughly after each addition, until a portion

*Not every bleaching powder makes a satisfactory solution for this test. Laundry bleach solution should not be used.

of the filtered bleach solution gives only the faintest precipitate of calcium carbonate upon addition of a few drops of the sodium carbonate solution. There should be no material quantity of the calcium salt left in solution, and the solution should be as neutral as possible. ( Excessive alkalinity militates against the success of the test. ) When this point has been attained, filter the calcium carbonate and make up the filtrate to one litre with water. Determine the available chlorine content of this solution either by standard sodium thiosulphate or arsenious acid solution. Then carefully dilute t6 3.00 ± 0.05 percent available chlorine content.

b) *From sodium hydroxide* — Prepare an approximately 1.5 N sodium hydroxide solution ( reagent quality ) in water, transfer to a narrow-mouthed amber coloured bottle and cool in an ice-bath. Bubble chlorine gas, freed from acid fumes by washing with water, through the caustic solution. Continue chlorination till about 2 ml of the liquor, when tested with one drop of phenolphthalein, gives a distinct pink colouration that persists but for not more than 5 seconds. After this point has been reached, determine the available chlorine content of this solution either by standard sodium thiosulphate or arsenious acid solution. Then carefully dilute to 3.00 ± 0.05 percent available chlorine content.

**26.1.3** *Procedure*

**26.1.3.1** Roll the 'prepared sample' ( *see* **3.3.2** ) to ensure uniformity and weigh 37.5 ± 0.1 g directly from the rolling sheet. Transfer to a 400-ml tall form beaker, add 3.7 ± 0.1 g of sodium carbonate and 110 ml of water. Place the beaker in a boiling water bath and stir with a variable speed electric stirrer. The solution will foam very strongly during the first few minutes. Beat down the foam by increasing the speed of the stirrer. Continue the stirring for exactly half an hour. Then remove from the bath, and add 25 ml of hot water ( about 70°C ) slowly and with stirring. Immediately strain the solution through a sieve of brass or copper having a nominal aperture of about 0.15 mm into a graduated cylinder, washing the beaker and the residue on the gauze with hot water ( about 70°C ) using a policeman, if necessary, until the volume is about 250 ml. Bring to a temperature of 27 ± 2°C and make up to 280 ml with water at 27 ± 2°C. Pour and drain into a 400-ml beaker and add slowly and with stirring 95 ml of bleach liquor. Stir occasionally for 30 minutes and allow to stand covered in a cool place overnight.

**26.1.3.2** Next morning, after first stirring in any scum which may have come to the top of the liquid by a gentle rotatory motion with a stirring rod dipped slightly below the surface and taking care not to disturb the sediment ( if any ) in the lower part of the solution, carefully measure into a tall form beaker 300 ml of the supernatant liquid. The decanted portion is

equivalent to 30 g of the seedlac taken and 75 ml of bleach liquor. Add with stirring 4 ml of bleach liquor and let stand for 30 minutes.

**26.1.3.3** Fit a 12.5-cm filter paper ( Whatman No. 1 or equivalent ) into a glass funnel and filter the bleached solution obtained in **26.1.3.2** through the dry filter paper. Reject the first 2 or 3 ml of the filtrate. Use the subsequent clear filtrate in a pre-calibrated photo-electric colorimeter in conjunction with a filter of spectral range 480 mμ for determining bleach index as indicated in **26.1.3.4.**

**26.1.3.4** Fit the instrument with the appropriate glass cell and the filter. Take sufficient distilled water in the cell and set to null point. Replace the distilled water with the bleached lac filtrate, adjust to null point again and note the reading on the indicator scale. Read the bleach index of the sample from the calibrated graph drawn as described in **26.1.3.5.**

**26.1.3.5** The colorimeter may be calibrated as follows:

Prepare N/200, N/400, N/500, N/750, N/1 000, N/2 000, N/3 000, N/4 000, N/5 000 and N/6 000 solutions of iodine in potassium iodide by diluting a standard N/10 solution. Fit the colorimeter with a glass cell of suitable effective depth and the selected filter. Take sufficient distilled water in the cell and set to null point. Replace the distilled water with N/200 iodine solution ( after rinsing the cell at least twice with that solution ) and set again to null point. Note the reading on the scale. Repeat the process using the remaining solutions one after the other. Draw a graph on semilog paper plotting the scale readings against bleach indices whose equivalents in terms of the iodine solutions are given below:

| *Strength of Iodine Solution* | *Equivalent Bleach Index* |
|---|---|
| 1/200 | 170 |
| 1/400 | 152 |
| 1/500 | 144 |
| 1/750 | 133 |
| 1/1 000 | 122 |
| 1/2 000 | 98 |
| 1/3 000 | 81 |
| 1/4 000 | 70 |
| 1/5 000 | 62 |
| 1/6 000 | 58 |

Once calibrated, this graph can be used for a long time for bleach index determinations with the same instrument. Its validity needs checking only occasionally.

## 26.2 Method II ( for Bleachability )

**26.2.0** *Outline of the Method* — Bleachability is determined by adding the specified number of millilitres of 3 percent available chlorine bleach liquor to 30 g of the seedlac sample. When all the bleach liquor is consumed the colour of the sample is compared with the standard sample and reported as lighter, equal to or darker. The amount of bleach liquor to be added for each grade of seedlac is specified as follows:

*Grade I*

| | |
|---|---|
| Class A | 80 ml |
| Class B | 100 ml |

Colours are to be compared to Grade I standard.

*Grade II*

| | |
|---|---|
| Class A | 115 ml |
| Class 8 | 140 ml |

Colours are to be compared to Grade II standard.

**26.2.1** *Reagents*

**26.2.1.1** *Sodium carbonate* — anhydrous, analytical reagent grade.

**26.2.1.2** *Bleach liquor* — as prescribed under **26.1.2.3.**

**26.2.1.3** *Standard seedlac for bleach tests* — A sample of seedlac of known bleachability obtained from a source recognized for the purpose, which, when treated with the appropriate quantity of bleach liquor, will give a bleached lac equivalent to N/4 000 iodine solution.

**26.2.2** *Procedure*

**26.2.2.1** The standard sample of Grade I or Grade II, whichever is appropriate to the unknown sample under test, shall be used for comparison, and shall be tested at the same time and under the same conditions of duration, temperature, etc.

**26.2.2.2** Samples for test against the standards for Grade I and Grade II shall be prepared by grinding the 'prepared sample' ( *see* **3.3.2** ) to pass entirely through 2.0-mm IS Sieve. Roll the ground sample on paper to ensure uniformity, and weigh 37.5 ± 0.1 g directly from the rolling sheet. Transfer the seedlac to a 400-ml tall beaker, add 3.7 ± 0.1 g of sodium carbonate and 100 ml of water at 27 ± 2°C. Place and fasten the beaker in a boiling water bath and start stirring with a variable speed electric stirrer. Lac generally foams very strongly within the first three minutes of stirring, so the stirrer speed has to be increased during this period to keep down foaming. When

it subsides, the stirrer is reduced to normal speed. After exactly 30 minutes of stirring, remove the beaker from the bath and add, slowly with stirring, 25 ml of hot water ( about 70°C ). Immediately strain the solution through 150-micron IS Sieve of brass or copper into a 500-ml graduated cylinder, washing the beaker and residue on gauze with hot water ( about 70°C ), using a policeman, until the total volume is about 200 ml or over but not exceeding the volume stated in col 3 of Table 1. Place the cylinder in a tall pail into which cold water is flowing and cool to 27 ± 2°C with stirring and then make up to the volume stated in col 3 of Table 1, with water at 27 ± 2°C. Drain into a 400-ml beaker. Immediately add, slowly and with stirring to the respective grades under test, appropriate volume of bleach liquor given in col 4 of Table I.

**TABLE 1 ADDITION OF BLEACH LIQUOR FOR BLEACHABILITY TEST — METHOD II**

| GRADE | CORRESPONDING STANDARD* | VOLUME TO CONTAIN 37.5 g or SAMPLE | VOLUME OF BLEACH LIQUOR TO BE ADDED | ALIQUOT TO BE TAKEN FOR TEST | VOLUME EQUIVALENT OF BLEACH LIQUOR | ADDITIONAL VOLUME OF BLEACH LIQUOR TO BE ADDED FINALLY |
|---|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| | | | | | | ml |
| Grade I: | | | | | | |
| Class A | I | 280 | 95 | 300 | 76 | 4 |
| Class B | I | 255 | 120 | 300 | 96 | 4 |
| Grade II: | | | | | | |
| Glass A | II | 236 | 139 | 300 | 111 | 4 |
| Class B | II | 205 | 170 | 300 | 136 | 4 |

*Standard I or II as appropriate.

**26.2.2.3** After adding the standard bleach liquor in accordance with the schedule for each grade, the total volume for all samples and standards shall be 375 ml. Stir the samples occasionally for 30 minutes, put a cover glass and allow to stand overnight at a temperature of 27 ± 2°C.

**26.2.2.4** After standing overnight, stir all the samples gently to distribute the wax which collects at the top, but immersing the stirring rod just below the surface, and taking care not to disturb the sediment. After distributing the wax, take out an aliquot of 300 ml for the test, as given in col 5 of Table 1. This volume represents 30 g of seedlac. To each of the solutions prepared above, add the additional volume of 4 ml of standard 3 percent bleach liquor, as given in col 7 of Table 1. Thirty minutes after this final small addition of bleach liquor, compare the samples under test with the appropriate standard, and report as lighter, equal to, or darker than the standard.

## 27. DETERMINATION OF PARTICLE SIZE OF SEEDLAC

**27.0 Outline of the Method** — A known mass of the sample is sieved through 600-micron IS Sieve and the mass of the material passing through is noted. The portion of the material passing through 600-micron IS Sieve is then passed through 425-micron IS Sieve and the fines are weighed.

### 27.1 Apparatus

**27.1.1** *Sieves* — one 600-micron IS Sieve and one 425-micron IS Sieve.

**27.2 Procedure** — Take approximately 100 g of the 'original observation sample' ( *see* **3.3.4** ) and break down loose lumps, if any, by gentle rubbing between fingers. Weigh accurately, and sieve in 600-micron IS Sieve with a gentle rocking motion till nothing more passes through. Collect the entire material passed through and weigh accurately ( $M_1$g ). Then transfer it to a 425-micron IS Sieve and continue sieving till nothing more passes through. Collect the entire material passed through and weigh accurately ( $M_2$g ).

### 27.3 Calculation

a) Material passing through 600-micron IS Sieve, percent by mass = $\frac{100\ M_1}{M}$

where

$M_1$ = mass of the material which passes through 600-micron IS Sieve, and

$M$ = mass of the material taken for the test.

b) Material passing through 425-micron IS Sieve, percent by mass = $\frac{100\ M_2}{M}$

where

$M_2$ = mass of the material which passes through 425-micron IS Sieve, and

$M$ = mass of the material taken for the test.

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